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CARBONACEOUS FIBER COMPOSITES

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UNITED STATES NAVAL ORDNANCE LABORATORY, WHITE OAK, MARYLAND

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ABSTRACT: Carbon and graphite fibers are now available with a range of physical properties; the graphite fibers are stiffest and strongest with moduli to 37×10^{11} dynes/cm^2 (54 million psi) and strengths to 28×10^9 dynes/cm^2 (400,000 psi). A total of 17 types of carbonaceous fibers was bonded with epoxy resins into unidirectional composites and tested for physical properties. Composite moduli and tensile strengths showed effective translation of fiber properties to composite properties, but shear strengths were low, ranging from 2.0 to 4.8 x 10^8 dynes/cm^2 (2900 to 7000 psi). These low shear strengths generally limited flexural and compressive strengths to relatively low values. A fiber treatment to grow silicon carbide whiskers and deposits on the fibers has given shear strengths up to 12.4 x 10^8 dynes/cm^2 (18,000 psi), but with fiber tensile strength reduction of 5 to 70%. Long-term water exposure of stressed composites showed varying results, depending on fiber and exposure condition.

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Carbonaceous Fiber Composites

This report describes the results of tests of graphite fiber reinforced plastic composites. The intent is to determine the potential of such composites for use in Navy structures, but the results are applicable to all high performance structures. The first report on this work was issued in December 1964 (ref. (a)). Further information was given in papers presented in February 1966 (ref. (b)), October 1967 (ref. (c)), and February 1968 (ref. (d)). This report summarizes the papers and also presents new data obtained through May 1968. The work is continuing and further reports will be issued. The work was funded by the Naval Ship Systems Command under Task NOL-972/SHIP3.

Limitations in the accuracy and significance of the results are imposed by the poor quality of some of the fibers as obtained from the manufacturers and by our lack of a good method of directly determining composite void content. These limitations are not considered to be serious enough to significantly compromise the conclusions presented in this report.

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Captain USN
Commander

Albert Lightbody
ALBERT LIGHTBODY
By direction

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INTRODUCTION

Within the past several years, a new group of fibers for structural use--carbonaceous fibers--has become commercially available. These include both carbon fibers and graphite fibers in a wide range of strengths and moduli, but the most important of these has come to be the graphite fibers. These fibers have moduli of 37×10^{11} dynes/cm^2 (54 million psi) and specific gravities of 2.0 or less, giving specific moduli higher than any other known continuous fiber. Strengths are also high, with fiber tensile strengths up to 28×10^9 dynes/cm^2 (400,000 psi) now available. The future will bring still higher values; moduli to 69×10^{11} dynes/cm^2 (100 million psi) and strengths to 55×10^9 dynes/cm^2 (800,000 psi) have been demonstrated in the laboratory and eventually will be commercially available. The outstanding potential of graphite fibers as compared to other fibers thus is well founded.

Because of this potential, the Naval Ordnance Laboratory, and others in the materials field (refs. (a) - (g)) have investigated these fibers for composite reinforcement. High modulus, low density graphite composites are an important new group of materials for use in high performance structures, particularly those in which failure is stiffness controlled. Included are mine cases, torpedo hulls, deep submergence hulls, helicopter blades, and various beams, struts, panels, ribs and stiffeners in aircraft, spacecraft and submersibles. Also, graphite fibers, which are expected to be stronger at higher temperatures, could become extremely important for structures which get hot, such as the SST aircraft.

During this program, five carbon and graphite fibers were available for test. The objective was to determine the potential of carbonaceous fibers for deep submergence applications. This report describes the fabrication and testing of simple composites made from these fibers and draws conclusions as to their structural potential. Reference (a) is the first report of this work. That report concluded that there was a ".....strong possibility that carbon-base filaments can be used in structural applications" and led to a continuation of the work. This (second) report will show that high moduli and strengths in composites have been attained, a number of finishes or methods of increasing the bond of the resin to the fiber are under study, and water resistance of the composites can be good or bad depending on the fiber used. Overall, the results are promising and the work is continuing.

EXPERIMENTAL WORK

A. OBJECTIVE

As noted in the introduction, the objective of this work was to determine the potential of carbonaceous fibers for deep submergence applications. Such applications require survival in a compressive stress field and in a high pressure water environment. Important mechanical properties are, therefore, compressive modulus and strength, interlaminar shear strength, and permanence of these properties with water immersion. But underwater structures also will have brackets, fasteners, etc. so that tensile and flexural properties are also important.

B. METHOD OF APPROACH

The approach used in this work was to make unidirectional composites using carbonaceous fibers in organic resin matrices, usually epoxy resins. Sometimes the fibers were surface treated before bonding for comparison with untreated fibers. After fabrication, the composites were tested for some or all of the following properties: (a) tensile modulus and ultimate strength, (b) compressive modulus and ultimate strength, (c) flexural modulus and ultimate strength, (d) interlaminar shear strength, (d) transverse properties, (f) water absorption, and (g) specific gravity and fiber content. Flexural and shear strengths after water exposure up to one year also were determined for several composites.

C. MATERIALS

1. Fibers. Carbonaceous fibers presently are available in short lengths and as continuous yarn or roving with 720 or more individual filaments in a cross section. Each filament is 5 to 12 microns in diameter, may or may not be round, and generally is somewhat porous. Specific surface can reach 200 m^2/gm or higher, although for graphite fiber the value typically is around 1.0. Specific gravity is 1.4 to 2 compared to 2.25 for pyrolytic graphite. The shape and porosity result from the manufacturing process. Rayon has been the most common starting material, and these rayon filaments have ridges, striations, and non-round shapes resulting from the viscose solution being extruded into the coagulating bath during the manufacturing process. Polyacrylonitrile also is used, and this gives a rounder fiber. To make carbonaceous fibers, these filaments are exposed to temperatures of 820°C (1500°F) or above in an inert atmosphere. This results in thermal degradation of the molecule with loss of up to 75% of the original weight as evolved gases. The carbonaceous residue that is left is the desired fiber and is 87 to 99+% carbon. Other starting materials, including polybenzimidazole, are being tried. These materials have a higher amount of carbon in the molecule than cellulose and so should give less weight loss and less fiber porosity after pyrolysis. Higher temperatures, to 1650°C (3000°F), give higher carbon contents, an increasingly graphitic crystal structure, and fibers with the highest moduli and strengths in the fiber direction. Anisotropy increases, however, so that, unlike glass, properties in the cross fiber direction are thought to be much lower than along the fiber. The consequences of this in structural composites use are as yet unknown.

The fibers, as now purchased from suppliers, present certain problems not encountered with glass fibers. Some fibers are good atmospheric scavengers and may absorb 12% of their weight or more in coming to equilibrium with room ambient air. This interferes with composite bonding, and the gases should be desorbed before the fibers are used. The fibers are very vulnerable to abrasion damage. It is typical that many breaks occur when filament winding is done. One manufacturer coats the fibers with polyvinyl alcohol to reduce abrasion vulnerability, but the general picture is still that all carbonaceous fibers must be handled gingerly. The following table lists the fibers tested in this study:

TABLE I

FIBERS TESTED

Amount Obtained	Type	Reported Fiber Modulus 10^{11} dynes/cm^2	Fiber Designation and Manufacturer	
1 pound	Carbon	4.1 (6 million psi)	Fluton 6311-L-1061	3 M Company
1 pound	Carbon	4.1 (6 million psi)	Fluton H 31	3 M Company
1 pound	Carbon	4.1 (6 million psi)	WB-70 2/0	Union Carbide Corp.
4 pounds	Carbon	4.1 (6 million psi)	VYB Roving	Union Carbide Corp.
1 pound	Carbon	4.1 (6 million psi)	CY 1604	HITCO
1/4 pound	Graphite	low	10% silicon carbide (coating)	Carborundum Corp.
1/4 pound	Graphite	low	30% silicon carbide (coating)	Carborundum Corp.
1 pound	Graphite	low	GSGY 2-5	Carborundum Corp.
1 pound	Graphite	low	WYB 125 1/5	Union Carbide Corp.
1 pound	Graphite	17 (25 million psi)	HMG 25	HITCO
2 pounds	Graphite	17 (25 million psi)	Thornel 25	Union Carbide Corp.
3 pounds	Graphite	28 (40 million psi)	Thornel 40	Union Carbide Corp.
2 pounds	Graphite	34 (50 million psi)	Thornel 50	Union Carbide Corp.
2 pounds	Graphite	26 (38 million psi)	RAE fiber	Royal Aircraft Establishment (Eng.)
2 pounds	Graphite	37 (54 million psi)	RAE fiber	Royal Aircraft Establishment

TABLE I (continued)

Amount Obtained	Type	Reported Fiber Modulus 10^{11} Dynes/cm^2	Fiber Designation and Manufacturer	
1 pound	Graphite	26 (38 million psi)	Morganite II	Morganite Ltd. (England)
1 pound	Graphite	37 (54 million psi)	Morganite I	Morganite Ltd. (England)

Some of these fibers were in early production at the time we obtained them and quality was poor. Some fibers had weak spots or stiff spots along the length; some were discontinuous on the spool; some would not off-wind from the spool without snagging and breaking. All of these factors caused winding problems and degraded the strengths of the composites made from them. Expected increasing use of these fibers in the future will result in more production and better quality control, thus minimizing the problems encountered with these first batches.

2. Fiber Treatments. Several surface treatments of the fibers were tried in attempts to increase the bond of the resin to the fibers, as measured by interlaminar shear strength. Critical surface tension for wetting of graphite was measured as 46 dynes/cm, compared to 35 dynes/cm for glass. Since epoxy resins have surface tensions of 44 dynes/cm, or less, it is seen that the graphite should be much more readily wetted than the glass. But test results (see "Results") showed low interlaminar shear strengths for untreated graphite fiber.

Because of the low shear strength, several graphite surface treatments were tried, as follows:

a. Whiskering. "Whiskering" is the name we applied to a process in which silicon carbide spikes or whiskers are "grown" on the surface of the graphite filaments. These whiskers are tiny, only 1 to 2% of the fiber diameter, and constitute 1 to 5% of the weight of the fiber. The theory is that these whiskers provide mechanical interference against the fibers being pulled out of the resin and thereby increase the interlaminar shear strength.

The whisker growing treatment is done by Thermokinetic Fibers, Inc. (TKF). They expose short-length bundles of fibers to a special atmosphere at 1100 - 1650°C (2000 - 3000°F). At these temperatures, the bundles are quite permeable to the gases used and whiskers grow readily on fibers and in spaces within the bundles. The fibers treated were the following: Thornel 25, Thornel 40, Thornel 50, HITCO HMG, SAMCO 320, RAE high modulus, and Morganite II. After treatment, the fibers were bonded into composites and tested for various properties.

b. Nitric Acid Treatment. This treatment, first described by Herrick (ref. (h)), consists of boiling the fibers in 60% nitric acid for 24 hours, then washing thoroughly with water and drying. The treatment is intended to

create chemical bonding sites for resin by reaction of the fiber with nitric acid to give carboxyl (-COOH) and hydroxyl (-OH) groups on the fiber surface. Surface area of the fiber also is increased, and fibers lose from 0.2 to 2.0% of their weight in the treatment. Some investigation also was done by us as to the effect of varying the treatment time, with 4 to 48 hours being tried. The following fibers were nitric acid treated through the course of this program: Thornel 50, HITCO HMG, SAMCO 320, and Morganite I. After treatment, the fibers were bonded into composites and tested for various properties.

c. CASING Treatment. CASING, or Crosslinking by Activated Species of Inert Gases, was tried only a slight amount in this program, although future work with this method is expected to be important. CASING consists of exposing the fiber to an inert gas (argon, neon, etc.) at reduced (1 millimeter) pressure, then imposing a radio frequency field on the gas. The gas ionizes and glows and the gas molecules, in this charged and energetic state, contact the fiber surface and provide energy for reactions between materials on the fiber surface (occluded gases, H_2O, O_2 and polyvinyl alcohol). These reactions, loosely called "crosslinking," can give sites on the fiber surface for chemical bonding with resins and so should increase the strength of this bond. In this program, Thornel 25 was given the CASING treatment and then bonded into a composite and tested for shear strength.

d. Silica Treatment. A small amount of work was done to put a silica coating on graphite fiber by exposing the fiber to tetraethyl orthosilicate at 900°C (1650°F). Silica coatings ranged from 4.5 to 15% of the fiber weight. Thornel 40 and Thornel 50 were treated, then bonded into composites and tested for shear strength.

3. Resins. The resin most used as the matrix was Union Carbide's ERL 2256 with ZZL 0820 hardener in a 100:27 ratio, which is nominally stoichiometric. The hardener is an eutectic mixture of metaphenylenediamine and methylene dianiline. A few composites were made with anhydride cured epoxies and with polyesters and phenolics. Composite properties were the same or lower with these resins; these composites generally will not be further noted in this report.

D. IMPREGNATION PROCEDURE

Early work (ref. (a)) showed that a special impregnation procedure was necessary to desorb possible occluded volatiles on the fiber prior to bonding. If this were not done, at resin curing temperatures of 100 to 150°C (212 to 300°F), some volatiles would be expelled, interfering with the resin-fiber bond. A vacuum-temperature drying procedure was used, which was then followed by impregnation with the resin without re-exposure to the air.

This procedure was used mainly with the carbon fibers, which lost up to 12% by weight when dried. As the graphite fibers became available, it was found that these generally lost approximately 0.2% weight at 150°C (300°F). Several tests of these fibers using the vacuum-pressure impregnation, and also using a simple dip impregnation, gave no difference in results. Dip impregnation, using a vacuum to assure thorough wetting, became the standard method with the graphite fibers.

E. SPECIMENS MADE

The fibers, when received as continuous lengths, were wound into NOL rings in accord with ASTM 2291-65T and as described by Kinna (ref. (i)). The tension on the strands during winding was limited by the low breaking strengths of most of the graphite yarns. When necessary, heated ring molds were used to reduce resin viscosity and to keep the resin contents in the rings at the desired value of approximately 40% by volume. Before and during winding, the fibers were inspected for visual appearance. Although some types were quite uniform, others had stiff lengths along the fiber, breaks, splices, shipping abrasion damage, and fiber sticking to itself during off-winding. These conditions did not preclude these fibers from being chosen for test, but they did make handling more difficult and fabrication of composites more time consuming. All of the fibers were found to be extremely prone to abrasion damage so that even with careful handling techniques some breaks occurred.

Those fibers that were too weak to wind, or were supplied as short lengths, were molded into straight bars 0.635 x 0.318 x 15 cm (1/4 x 1/8 x 6"). Weighed amounts of fiber were tied into bundles; then these bundles were vacuum impregnated, the ties cut, and the bundles placed in a mold and cured. Completed bars were machined to length and tested.

F. TESTS

1. Strengths and Moduli. Graphite composite specimens were tested according to standard ASTM methods, if available, or by procedures developed in this work. Table II lists the tests used:

TABLE II

COMPOSITE SPECIMENS AND TESTS

Test	Specimen	Method
Ultimate tensile strength	(1) individual filament (2) strand (3) NOL ring 0.152 cm wall (0.060" wall)	Method 1* ASTM D2343-65T Reference (i)
Tensile modulus	(1) NOL ring 0.152 cm wall (0.060" wall)	Reference (i)
Ultimate compressive strength and compressive modulus	(1) NOL ring 0.318 cm wall (0.125" wall)	Reference (i)
Ultimate flexural strength and flexural modulus	(1) NOL ring section 16:1 span/depth (2) Straight bar 16:1 span/depth	Ref. (i)
Interlaminar shear strength	(1) NOL ring section 5:1 span/depth (2) Straight bar 5:1 span/depth	ASTM D2344-65T; also Reference (m); also guillotine test

TABLE II (continued)

Test	Specimen	Method
Transverse properties	(1) Flat plate 7.6 x 33 x 0.25 cm (3 x 13 x 0.098")	Method 2*

* For a description of methods 1 and 2 see Appendix A.

2. Long-Term Water Exposure. As an indication of deep submergence applicability, composites made with Thornel 25, RAE 54 (Morganite I), and low modulus carbon fibers were exposed to water and then tested for strength retention. The procedure used was to break a group of control specimens in flexure and shear, then to stress the remaining specimens to 0%, 50%, or 70% of the control breaking loads by using a fixture like the one shown in Figure 1. These stressed specimens, in their fixtures, were then placed in one of several environments, which included ambient room conditions, water immersion, and immersion in water at 4000 psi. Withdrawals were made after three weeks, 13 weeks, and one year exposure. After removal from the test conditions, the specimens were broken in flexure or shear and their strengths compared to the control values.

3. Multiaxial Fatigue. Low modulus (4.1×10^{11} dynes/cm^2, or 6 million psi) composites were tested according to the multiaxial fatigue method developed by Kinna and Prosen (ref. (j)). This test consists of cyclic torsion of a half ring and mainly measures the retention of the torsional rigidity and torsional strength. Higher modulus graphite fiber composites having low shear strengths were not tested. The test was run by taking control specimens and twisting them until they broke, then taking additional specimens (minimum of 5 replicates) and twisting them to either 50% or 70% of the breaking rotation for 10,000 cycles both in air and in water. During cycling, the torsional rigidity is continuously recorded. After 10,000 cycles, the half rings are twisted to break, and the loads and angles recorded.

4. Fiber/Resin Determinations. Several methods were evolved to determine the fiber content of carbonaceous composites. Standard methods of burn-off used with glass composites do not work because carbonaceous fibers oxidize. None of the methods developed is considered to be ideal for all fibers in that they may take too much time, are not accurate enough, or are not compatible with certain fibers. A listing of the methods follows:

a. Thermogravimetric Analysis. A procedure in which the temperature of the sample is slowly raised (in a vacuum) and the weight change (due to pyrolysis) is continuously monitored. The weight remaining at elevated temperature is the fiber and the resin residue. From separate runs on fiber only and resin only, data are obtained to calculate the composite resin/fiber composition. A single determination takes four hours, the equipment is relatively expensive, and the answer is accurate to an estimated ±2%. Reference (b) gives more complete information.

b. Heating in Inert Atmosphere. Requires 24 hours time at a temperature of 800°C (1470°F) in a dry argon atmosphere. It is probably the most

satisfactory all around method. The fibers are essentially unaffected and the resins lose 80 to 92% of their weight.

c. Heating in Air. A very useful technique for some fibers and uses simple equipment. At 420°C (790°F) for 15 1/5 hours the resins lose 99.6% of their weight and fibers lose from 0 to 2%. Excluded is Morganite II fiber, which loses too much weight.

d. Nitric Acid Digestion. Resins lose 100% of their weight and fibers lose 1/2 to 5%. Analysis is time consuming.

e. Sulfuric Acid Digestion (followed by sodium hydroxide reflux). The fibers may lose essentially no weight in this procedure, but it is extremely time consuming.

For fiber determinations in this work, method (c) was used most, with methods (a), (b), (d) and (e) used sparingly or in exploratory ways.

RESULTS

A. CARBON FIBER COMPOSITES--STRENGTHS AND MODULI

1. Tensile, Compressive and Flexural Strengths. Five carbon fiber composites gave results as shown in Figure 2. The Union Carbide VYB fiber composites proved to be strongest, with an average tensile strength of 6.45×10^9 dynes/cm^2 (93,500 psi) and a compressive strength of 10.7×10^9 dynes/cm^2 (155,000 psi). This proved to be the highest compressive strength measured for any composite throughout this program. Fiber contents for these composites ranged from 52 to 78% by volume; the calculated fiber stresses for the VYB composites were 10.7×10^9 dynes/cm^2 (155,000 psi) tensile and 18.0×10^9 dynes/cm^2 (260,000 psi) compression.

2. Interlaminar Shear Strengths. Five carbon fiber composites gave results as shown in Figure 3. The highest value was 9.30×10^8 (13,500 psi) again with Union Carbide VYB fiber. This shear strength is quite high and compares with the best glass fiber composites. Six-hour boils generally reduced the strengths only slightly, a very refreshing result to those who remember the results of boiling the first glass fiber composites.

3. Moduli. The moduli of these composites are relatively unimportant. The fibers have low moduli, and composites therefrom would not be expected to be used on a modulus basis. However, composite tensile moduli were measured and ranged from 2.0 to 3.3×10^{11} dynes/cm^2 (3.0 to 4.8 million psi).

B. GRAPHITE FIBER COMPOSITES--STRENGTHS AND MODULI

1. Mechanical Strengths in Fiber Direction

a. Tensile Strengths. The fiber strengths from strand and NOL ring tests of seven graphite composites are shown in Figure 4. Composite strengths for NOL rings also are indicated. Fiber strengths in strands ranged from 9.3 to 21.1×10^9 dynes/cm^2 (135,000 to 306,000 psi). Fiber strengths developed

in NOL rings were very good, averaging 85% of the filament strengths. By comparison, S-994 glass fiber NOL rings typically give only 60% of the filament strengths. The effective development of the graphite fiber tensile strength in composites is an important asset to these fibers.

b. Compressive and Flexural Strengths. The fiber strengths from ring and bar tests of the seven graphite composites are shown in Figure 5. Composite strengths also are indicated. Fiber strengths ranged from 6.8 to 16.5 x 10^9 dynes/cm^2 (99,000 to 240,000 psi). These results generally were considerably lower than the tensile values because the specimens nearly always failed in a buckling mode, resulting from low shear strength. The relationship between shear strength and compressive strength has been well documented (refs. (k) and (l)). For these composites, increases in compressive and flexural strengths will require increases in shear strengths.

2. Mechanical Strengths--Transverse to Fiber Direction. A limited amount of transverse strength data were obtained, mostly in conjunction with the whiskered fibers. Figure 6 shows test results of whiskered graphite fiber composites and indicated that transverse properties increased appreciably with whiskering. At the maximum whiskering amount, 3.4%, transverse tensile, flexural, and compressive strengths were at least double the assumed values for unwhiskered composites. The unwhiskered composites generally were so weak in transverse properties that they were difficult to fabricate, machine, and test. The values obtained were questionable and so were not included herein. The whiskered composites tested did not go to high enough whisker concentrations; it is expected that increasing the whiskering above 3.4% will give still higher transverse values. This investigation is continuing.

3. Interlaminar Shear Strengths

a. As-received Fiber. As-received fiber strengths from short beam tests of ring and bar specimens are shown in Figure 7. The low strengths are apparent, averaging only 2.9 x 10^8 dynes/cm^2 (4,200 psi) for the seven composites tested, and with a high value of 4.8 x 10^8 dynes/cm^2 (7,000 psi) for the Morganite II composite. The values dropped relatively little as a result of a six-hour boil, but all are so low as to be considered the achilles heel of graphite fiber composites. Such composites would fail quickly under appreciable compression, shear, or torsion loads.

b. Whiskered Fiber. As noted in the section on "Materials," some fibers were given a treatment to "grow" silicon carbide whiskers on the fibers and thereby give mechanical interference against pulling out of the resin. Figure 8 shows a single fiber that was whiskered. Growths could also be more or less than this, depending on exposure time. When fibers were treated in bundles, they looked different. Figure 9 shows a fiber taken from a whiskered bundle, and the so-called whiskers now look more like lumps or plating. But Figure 10 is an electron silhouette photomicrograph of the plating, and at magnifications up to X137,000 the plating is seen to be more of a moss or wool. The silicon carbide growth on the fibers is therefore a combination of whiskers, lumps and wool. Such treated fibers, when bonded into composites, gave shear strengths as noted in Figure 11. Indicated is a large increase in shear strength. By using the short beam method, shear strengths usually were so high that the specimens broke in flexure, rather than shear. Because of this, two

additional types of shear tests were run. A scissors or guillotine test gave results which showed whiskering to increase the shear strength by a factor of four. A second shear test, developed by the University of Virginia (ref. (m)), also showed shear increases by a factor of four, with some values reaching a high of 12.4 x 10^8 dynes/cm^2 (18,000 psi). Schematics of the three shear test methods are shown in Figure 12.

Further evidence of the effect of whiskering on the bond is shown in Figures 13 - 15 (scanning electron photomicrographs). A good bond between resin and fiber is evidenced in Figure 13, which shows coherent bundles of fiber encapsulated by ample amounts of resin. A poor bond is shown in Figure 14, which is the sheared surface of an RAE composite. Loose fibers are lying about and little or no resin is seen. But this same fiber, after whiskering, gives a much better bond, as shown in Figure 15. Once again, the fibers are in bundles and resin is seen. Interpretation of these pictures is taken from information in reference (n). By shear strength measurements and photograph evidence, the whiskering was shown to be extremely effective in increasing the shear strength of graphite composites.

The process, however, has the drawback of reducing the tensile strength of the fibers. Figure 16 shows the effect of "light" to "heavy" whiskering on strand and filament tensile strengths. The Thornel fibers are seen to be relatively resistive to degradation, at least up to "light-medium" whiskering, but the Morganites I and II and SAMCO 320 lost 25 to 50% of their strengths in the same treatment. This loss in strength is seen to be a major drawback but also may be indicative only of first attempts at whiskering. The process is new and some batches have been considerably better than others. Future work may result in more optimum treatment conditions.

c. Nitric Acid Treated Fiber. Treatment by nitric acid was a supplement to the work described by Herrick (ref. (h)). Figure 17 shows the results of treating four types of fibers and indicates increased shear strengths from 4% to 350%, with Morganite I giving 6.7 x 10^8 dynes/cm^2 (9750 psi) shear strength, the highest value of all tested. Thornel 50, treated for times varying from 4 to 48 hours, showed a maximum shear strength at 32 hours. Strand strengths of nitric acid treated material are lowered by 0 to 20%, with 10% being a common value. The method generally is reasonably effective, but the tedious treatment procedure is a drawback to extensive use.

d. CASING Treated Fiber. The exposure of Thornel 25 to the CASING treatment resulted in a 30% increase in shear strength to 3.3 x 10^8 dynes/cm^2 (4810 psi). This result, from the first attempt, is promising enough to warrant continued investigation.

e. Silica Treated Fiber. Fiber so treated gave from 20% less to 15% greater shear strength than for untreated fiber. The treatment is considered to be of no benefit in increasing shear strength.

4. Moduli. The moduli of these composites are probably their most important property; much of the future of graphite fiber is based mainly on their high specific moduli. These moduli were measured and are shown in Figure 18. Axial composite moduli ranged from approximately 9.7 x 10^{11} dynes/cm^2 (14 million psi) to approximately 28 x 10^{11} dynes/cm^2 (41 million psi). For a

single specimen, the composite tensile, compressive and flexural moduli all were different. Fiber moduli were not measured directly but rather were calculated from composite moduli and the law of mixtures. Fiber moduli calculated in this way generally gave lower values than the manufacturer's advertised values. The specific moduli of these composites, expressed as the composite modulus divided by the specific gravity, are shown in Figure 19; the high specific moduli of these graphite composites in comparison with other materials are apparent.

C. LONG-TERM WATER EXPOSURE

Results of the long-term water exposure tests are shown in Figures 20, 21 and 22. These figures are bar graphs, with each bar an average of two or more samples. Individual values usually did not vary more than 10% from the averages. These figures show a considerable difference among the three composites made from Thornel 25, RAE, and low modulus carbon fibers. The RAE composites survived both the shear and flexural tests fairly well, with significant strength drop-off shown only by flexural specimens in a wet environment under load for one year. This strength drop-off is as yet unexplained because it is unlikely the water attacked or affected the graphite fibers, and the shear results indicate the resin/fiber bond was unaffected.

The Thornel 25 composites were much more severely damaged, with both flexural and shear specimens generally breaking in the fixtures when exposed to a wet environment. These failures are speculated to be the result of resin-fiber bond failure promoted by the presence of the water soluble polyvinyl alcohol coating put on by the manufacturer of Thornel fibers to increase abrasion resistance. Thornel fibers without a PVA coating were not tested. The flexural specimens all failed by buckling on the compressive side, showing a shear failure rather than a fiber weakening. This shear strength degradation did not show up at all in six-hour water boil tests, which in the past have shown very well the susceptibility of glass fiber composites to water degradation.

Testing of a low modulus carbon fiber composite was included to determine the effects of the water environment on a high shear strength carbonaceous fiber. As shown in Figures 21 and 22, the results were varied, with no flexural strength loss but some shear strength loss for specimens which were stressed, and particularly for those immersed in water. As shown in Figure 20, these samples gained 11% in weight during the 52 weeks water exposure. This weight gain results from the hygroscopicity of the fibers in addition to the normal gain for the resin matrix. On fabrication into the composites, the fibers are dried and lost about 12% (mostly moisture) of their weight. After bonding, since most of the pores are too small for resin molecules to enter, the fiber can be assumed to be still somewhat hydroscopic and provides a ΔP for water flow into the composite until equilibrium is reached. This inflow of water is suspected to promote creep and then failure of the stressed samples. Unstressed samples exhibited little or no shear strength loss, showing that water pickup by itself is not necessarily deleterious when long-term loading is not a factor.

As concerns the test condition parameters, the 4000 psi water environment had only a little more effect than simple water immersion. Stressed samples failed much more quickly than unstressed, but there was no real qualitative difference between results from the 50% and the 70% stress levels. Fifty-two

weeks was unnecessarily long, with most results being adequately indicated by the end of thirteen weeks. Generally, the overall results are interpreted to mean that simple water immersion of composites under stress can show significant differences in the strength permanence of the composites. In this test, RAE composites were not damaged much, while the Thornel 25 and low modulus carbon composites did suffer considerable damage.

D. MULTIAXIAL FATIGUE TEST

This was a test of low modulus carbon fiber composites only and gave results as shown in Figure 23. These results indicate that the composites retained from 42 to 63% of their strength at 10,000 cycles, depending on the specific test conditions. Surprisingly, the condition which was expected to be mildest, a dry 50% twist, proved to be the most severe and gave the lowest values. This condition was repeated, giving ten replicates in all, with the same result. The conclusion from these data is that it makes little difference for a given composite whether the specimens are dry or wet or whether 50 or 70% twist is used. Our cycling rate was 30 cycles per minute so that a full test took 5 1/2 hours. This probably is not enough time for any significant water permeation. Overall, the retention of 42 to 63% of strength is considered good. Tests of other material, S glass and epoxy composites, for instance, usually give lower strength retentions, ranging from 15 to 50% (ref. (o)).

DISCUSSION

A. CARBON FIBER COMPOSITES

The low modulus carbon fibers were tested to give a more complete picture of these carbonaceous materials. It was recognized that their moduli and strengths generally would be lower than the values for graphite fibers. However, it is expected that at high temperatures, particularly when not in contact with oxidizers, the carbon fibers are expected to have many structural and insulating applications. High temperature testing was not within the scope of this work.

B. GRAPHITE FIBER COMPOSITES--STRENGTHS AND MODULI

1. Strengths. The strengths and specific strengths of graphite composites can be moderately high, but these are not yet up to the levels of the strongest materials, such as fiberglass. Manufacturers estimate graphite fiber tensile strengths over 34×10^9 dynes/cm^2 (500,000 psi) in a few years; laboratory batches to 55×10^9 dynes/cm^2 (800,000 psi) already have been made. Graphite composites thus have a potential to be among the highest in specific tensile strength.

2. Interlaminar Shear Strengths. The low interlaminar shear strengths measured in this program also have been reported by others (references (a), (b), (f), (g) and (h)) and improvement of this strength seems to be an objective of most people who work with these fibers. These low strengths may be caused by occluded gases on the fiber surfaces. The graphite crystal has unsatisfied valences at the surfaces and oxygen or water would react with the surfaces and

be bound very tenaciously, interfering with resin bonding. Heat cleaning the fiber prior to bonding has given increased shear strength, lending credence to this theory. It was also speculated that the transverse strengths of these highly anisotropic fibers may be so low as to result in peeling off the outer fiber layers as the mechanism of shear failure. This theory now seems less likely because if this were true, then no surface treatment would be likely to help, but in fact some of them do help. None of the methods noted in this report are considered to be fully satisfactory. The whiskering is considered to be quite significant, because, in addition to vastly increasing shear strength, it vastly increases transverse strengths. These are all aspects of the same phenomenon; i.e., better fiber-to-resin bond. But it is speculated that of all of the methods for improving this bond, whiskering may give the most enhancement of transverse properties. This work admittedly is incomplete and material surface treated in other ways needs to be tested in the transverse orientation. The main whiskering drawback is reduction of fiber tensile strength; the process was first attempted with graphite fibers in March 1967, so it is still quite new and is expected to improve in the future.

The nitric acid treatment, requiring a 24-hour boil, then repeated water washings, is unattractive because of the tedious procedure. Vapor treatments using gaseous oxides of nitrogen or other oxidizers might be developed to improve the processing.

The CASING treatment, tried slightly and giving a 30% increase in shear strength, may be something unique, or it in fact may be another way to either clean or oxidize the fiber surface. Further testing is intended.

Remaining for comment are the two manufacturers' proprietary treatments for Morganites I and II. The treatment doubles the shear strength of I, to 3.8×10^8 dynes/cm^2 (5,500 psi), and has not yet been tested on II. The manufacturer claims no tensile strength reduction. Details of the treatment are not known.

All in all, surface treatments to increase interlaminar shear strengths are in development, with none as yet clearly optimum.

C. FATIGUE TESTING

The two types of fatigue tests run here--static fatigue (or strength loss with time) and dynamic fatigue (or strength loss with cycles)--are separate aspects of the same problem--permanence qualities of the fibers and the fiber/resin bonds. For any future use of these composites, it is these permanence qualities rather than single-pull results from freshly made specimens which will be the criteria for design. The only fiber here to get both tests was the low modulus carbon, and, in spite of its outstanding initial shear strength, it showed general shear failure when stressed in water. The two types of tests are considered to complement each other and such testing is continuing.

D. PRICE

Price of the graphite fibers has not yet been mentioned in this report, but the present price is high enough to deserve some comment. The fibers, since introduction and up to recently, cost $500 to $550 per pound, about the price of

gold. There was a recent price reduction of a major U. S. fiber to $350 per pound, but English fibers are still $400 to $500 per pound, plus duty and shipping. These prices reflect the facts that the present fibers are still experimental and the processes for producing graphite fibers have not yet been reduced to a production basis. But the great potential of the fibers includes the fact that the price is expected to reduce considerably during the next few years, and a recent 20-year forecast (ref. (p)) projected a price of $3 - $40 per pound within 20 years. Such prices would make graphite fibers cost effective for many applications.

SUMMARY AND CONCLUSIONS

Carbon and graphite fibers are now available commercially in continuous lengths suitable for filament winding or in short lengths for hand lay-up. Their main advantages at present are high moduli, to 54 million psi, and low density, 2 gm/cm^3 or less, giving specific moduli higher than any other presently available material. There is also the very important potential of high temperature retention of strengths and moduli, provided the fibers are protected from oxidation.

Some of the fibers, as received, showed poor quality control and early production problems on the part of the manufacturers. Five low modulus carbon fibers and twelve graphite fibers were wound into NOL rings or molded into straight bars, using epoxy resin matrices. A vacuum-temperature-pressure impregnation procedure was used if the fibers lost weight on drying, or a vacuum procedure was used if they did not lose weight. The fibers were very vulnerable to abrasion and broke often in the winding and handling processes. Making the simple composites took 5 to 10 times as long as making the same items of glass fiber, showing possible problems for production use of the fibers.

The carbon fibers were of relatively less interest in this program and were tested mainly to give a more complete picture of carbonaceous fiber and composite properties. Tests showed composite strengths to range up to 10.7 x 10^9 dynes/cm^2 (155,000 psi) and moduli to 3.3 x 10^{11} dynes/cm^2 (4.8 million psi). Shear strengths were good, going as high as 9.3 x 10^8 dynes/cm^2 (13,500 psi).

Graphite fibers were the main interest. Tensile strengths of strands ranged from 11.0 to 21.0 x 10^9 dynes/cm^2 (160,000 to 306,000 psi), correlating reasonably well with the manufacturers stated filament strengths. Fiber tensile strengths in NOL rings averaged 65% of filament strengths, as good or better translation of strength properties than is obtained with S glass composites. Compressive and flexural strengths were limited by low shear strengths.

Interlaminar shear strength values varied widely depending upon the method of test. They were quite low, ranging from 1.9 to 4.8 x 10^8 dynes/cm^2 (2,800 to 7,000 psi) for composites made from as-received fiber and tested by the short beam method. Several fiber surface treatments were tried in attempts to increase the shear strengths. Of these, "growing" silicon carbide whiskers on the fibers by a special furnace treatment gave the highest shear values, 8.3 to 12.4 x 10^8 dynes/cm^2 (12,000 to 18,000 psi, as tested by the University of Virginia method), and also increased transverse strengths markedly. The treatment can reduce fiber tensile strength by 5 to 50% or more; the treatment is new and development is continuing. Other shear treatments tried include nitric acid boil of the fibers,

CASING exposure of the fibers, and a silica coating on the fibers. Investigation of the CASING treatment is continuing.

Graphite composite moduli ranged from 14 to 41 million psi in the fiber direction. Calculations of fiber moduli showed generally lower values than those advertised by the manufacturers. The highest values of specific composite moduli obtained in this program exceeded nearly all other structural materials; only beryllium is higher at the present time.

Composites exposed to long-term stress in water gave widely varying results. RAE composites survived reasonably well, but Thornel 25 and a low modulus carbon fiber composite had a high failure rate. It is theorized that fibers with water soluble coatings or fibers that are hygroscopic are likely to produce composites that are relatively vulnerable to water attack. A multiaxial (shear) cycling fatigue test (10,000 cycles in 5 1/2 hours) of a low modulus carbon fiber composite showed about 50% shear strength retention, which is good for this type of test. Multiaxial fatigue testing of high modulus composites awaits shear strength improvements.

The best fibers of those tested were found to be Morganites I and II and SAMCO 320. These exceeded the others in specific strengths or moduli and also were easiest to handle and use.

The results of tests of carbon and graphite fibers as presented in this report are interpreted as being generally favorable to the eventual structural use of these fibers. Specific strengths, as well as specific moduli, are expected within a few years to exceed the values for any other structural materials.

RECOMMENDATIONS

This study has pointed out several problems which bear further work, as follows:

1. The problem of low interlaminar shear strength still looms as the main task to be solved. Work toward its solution is recommended.

2. Different composites can show markedly different abilities to bear stress in water. Composite strength and modulus permanence properties need continued testing.

3. High temperature properties, considered to be a real forté of graphite fibers, need testing.

The above three items would appear to us to be the most significant at the present time as relating to eventual Navy structural use of these fibers.

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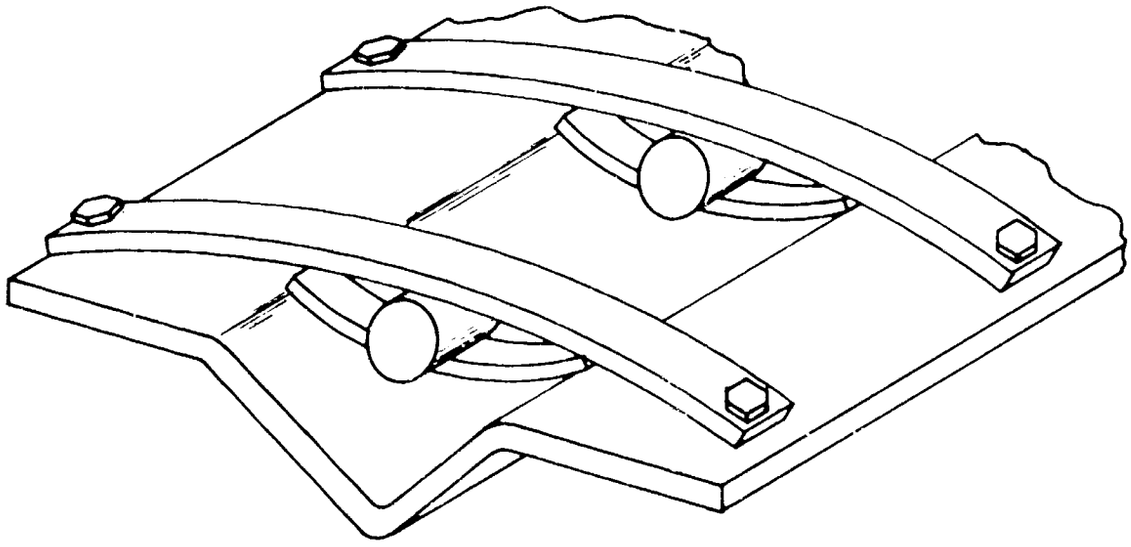


FIG. 1 FLEXURAL TEST FIXTURE FOR LONG-TERM LOADING
Composite Specimens Stressed to Predetermined Values by Bent Bars

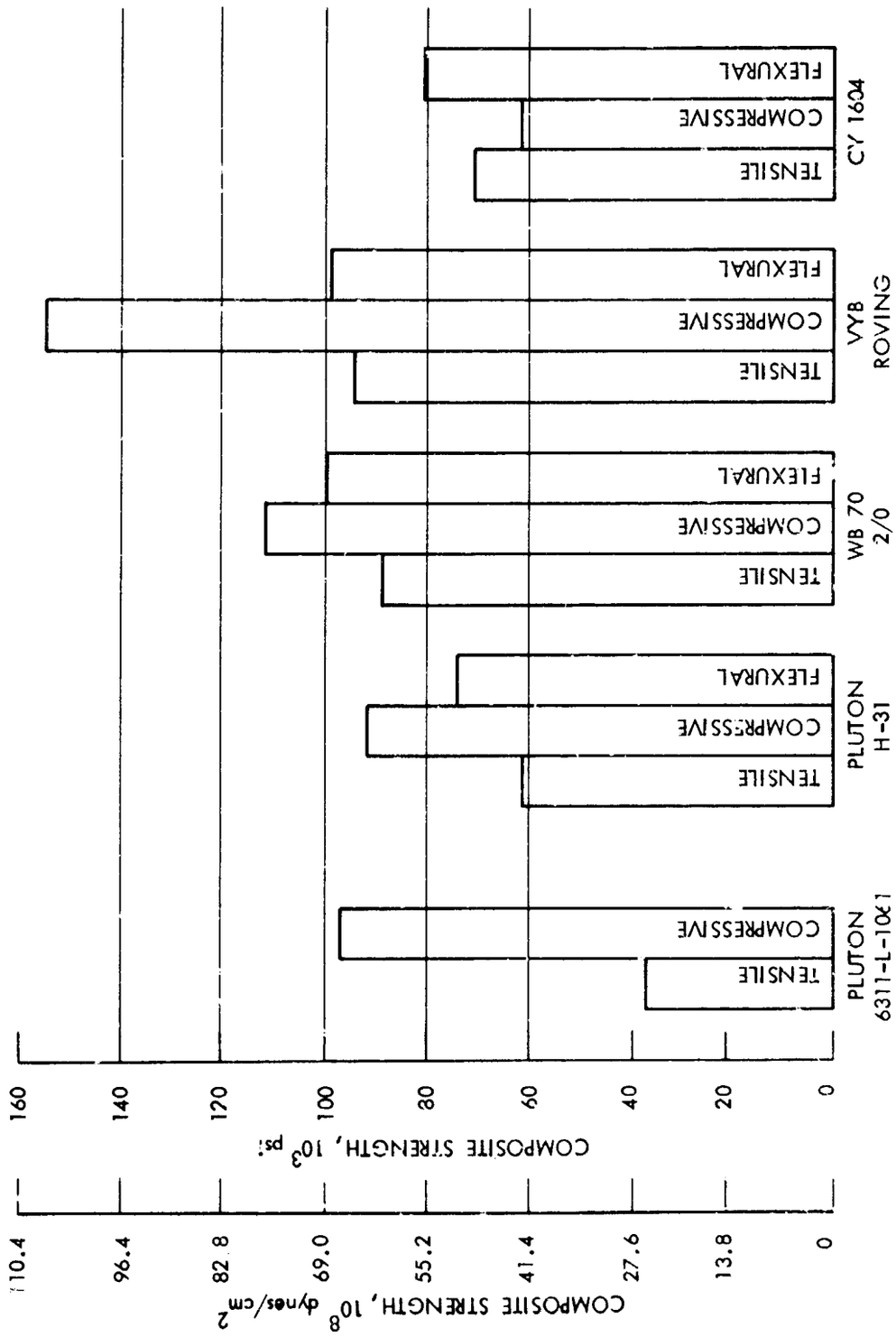


FIG. 2 COMPOSITE STRENGTHS
Low Modulus Carbon Fiber Composites
Average Values from NOL Rings

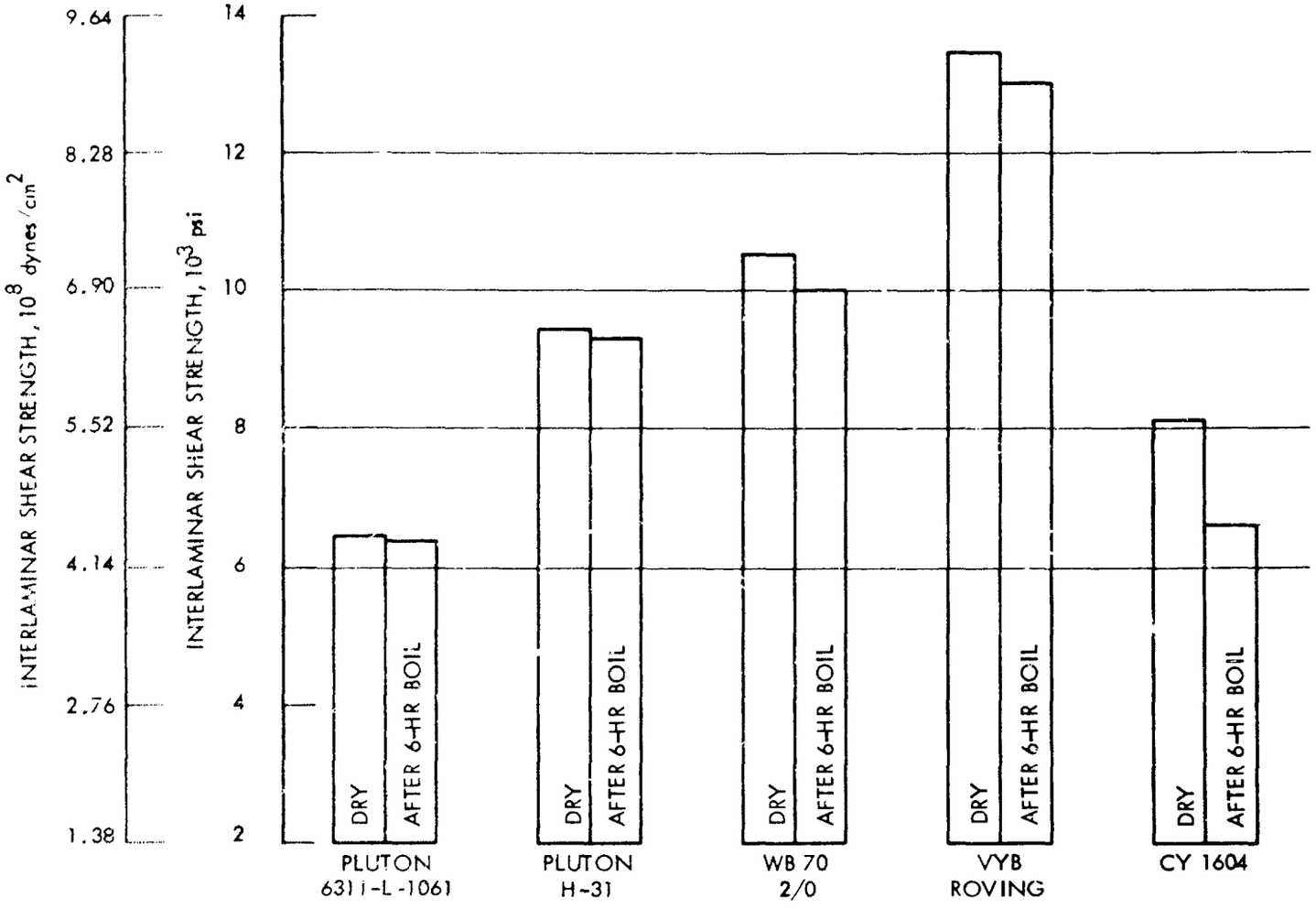
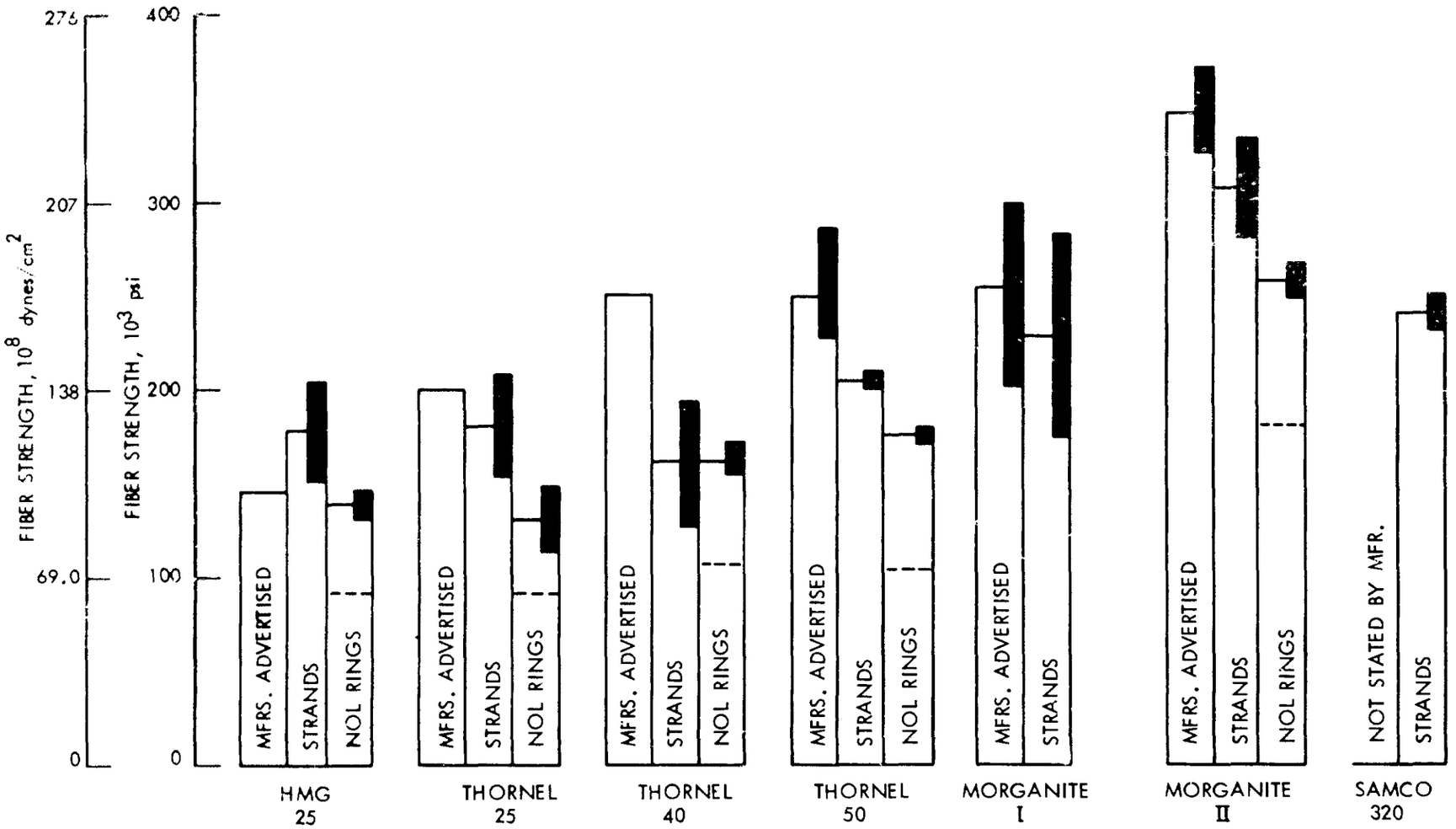
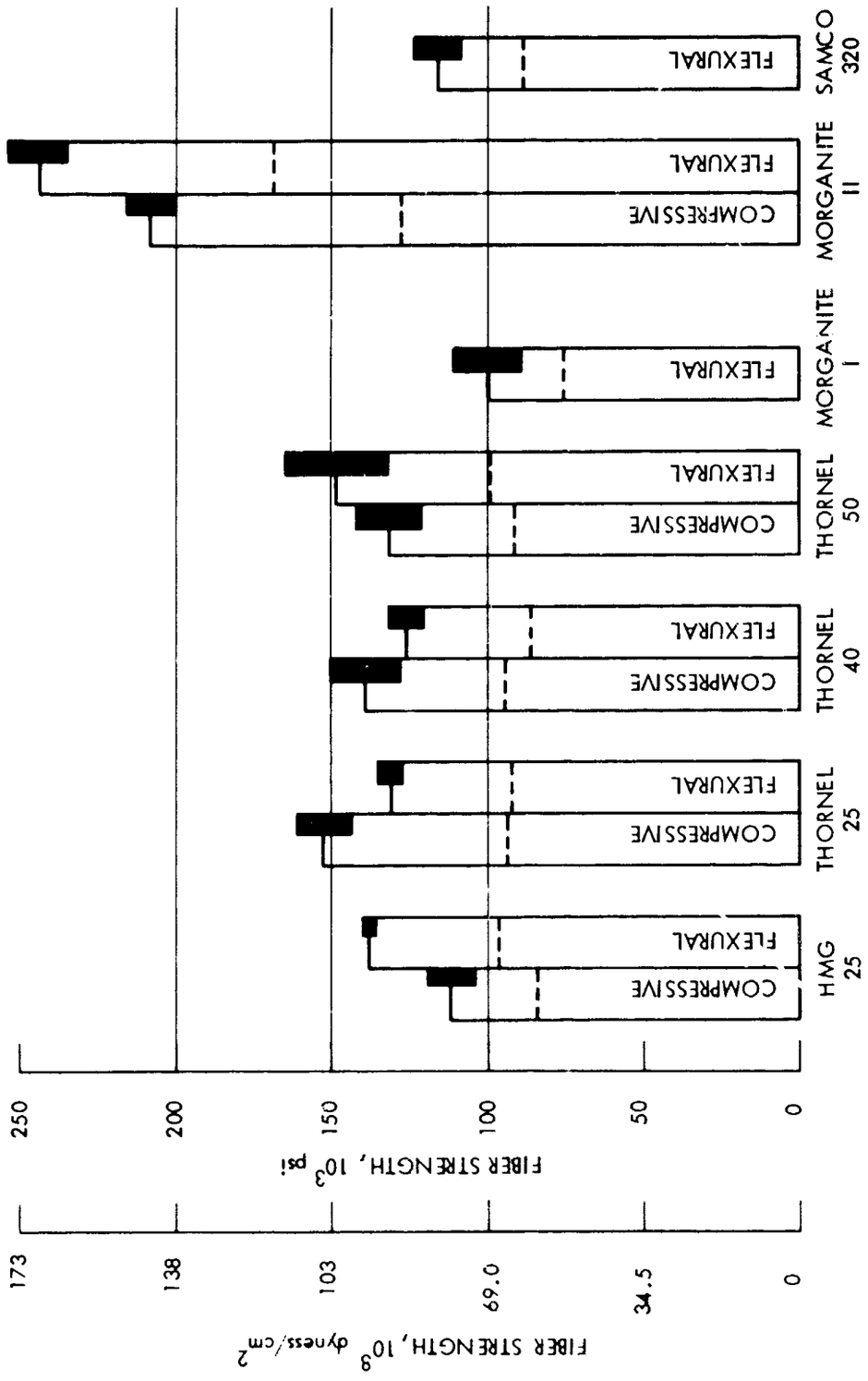


FIG. 3 INTERLAMINAR SHEAR STRENGTHS
Low Modules Carbon Fiber Composites Short Beam Method;
Average Values from NOL Ring Specimens



DOTTED LINES THROUGH "NOL RINGS" BARS INDICATE COMPOSITE STRENGTHS
SHORT DARK BAR SEGMENTS INDICATE THE RANGE OF VARIATION.

FIG. 4 GRAPHITE FIBER TENSILE STRENGTHS
Average Calculated Values from Tests of Strands and NOL Rings



DOTTED LINES THROUGH BARS INDICATE COMPOSITE STRENGTHS
SHORT DARK BAR SEGMENTS INDICATE THE RANGE OF VARIATION

FIG. 5 ULTIMATE FIBER STRENGTH
Average Calculated Values from Compressive and Flexural Tests
of Unidirectional Graphite Fiber Composites

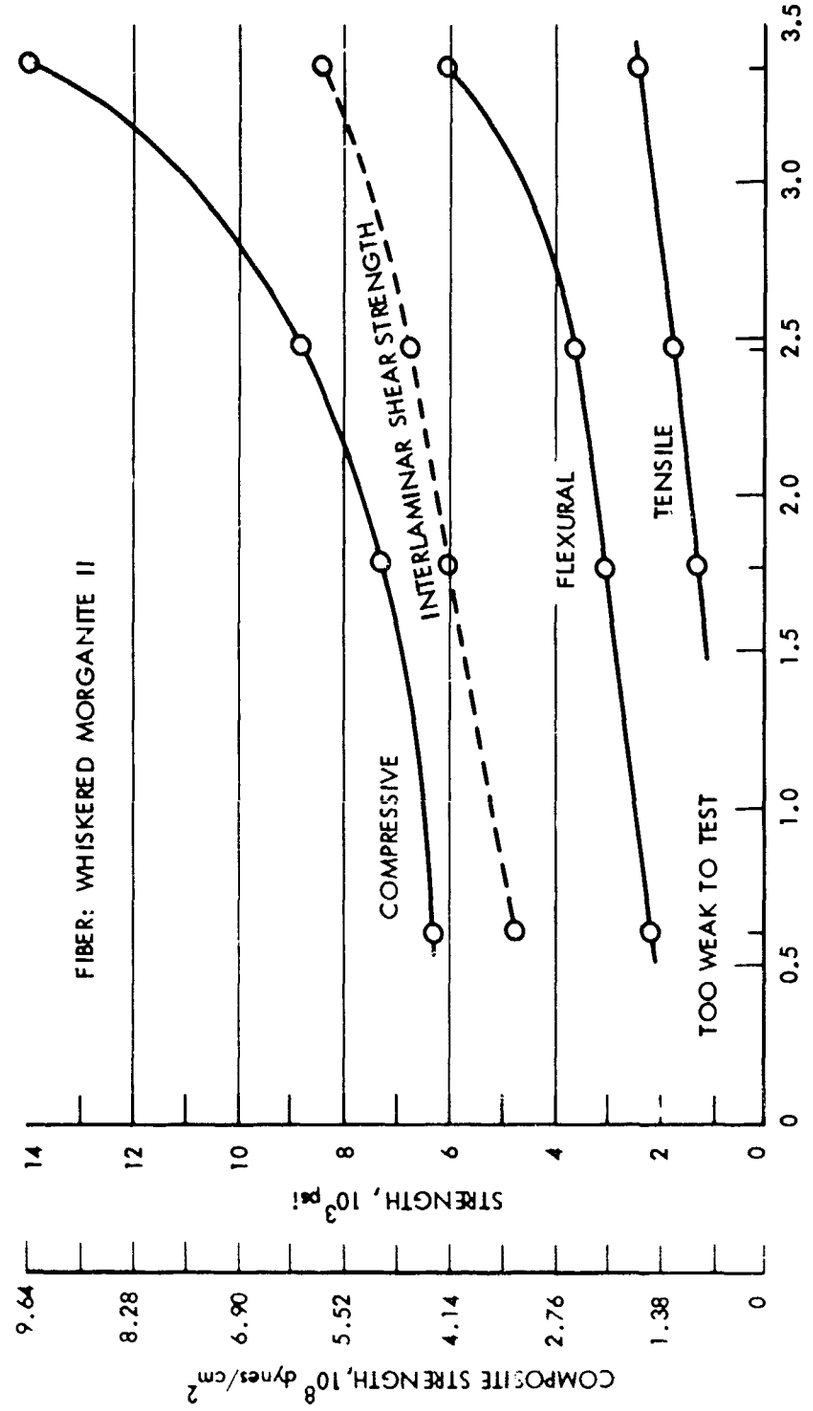
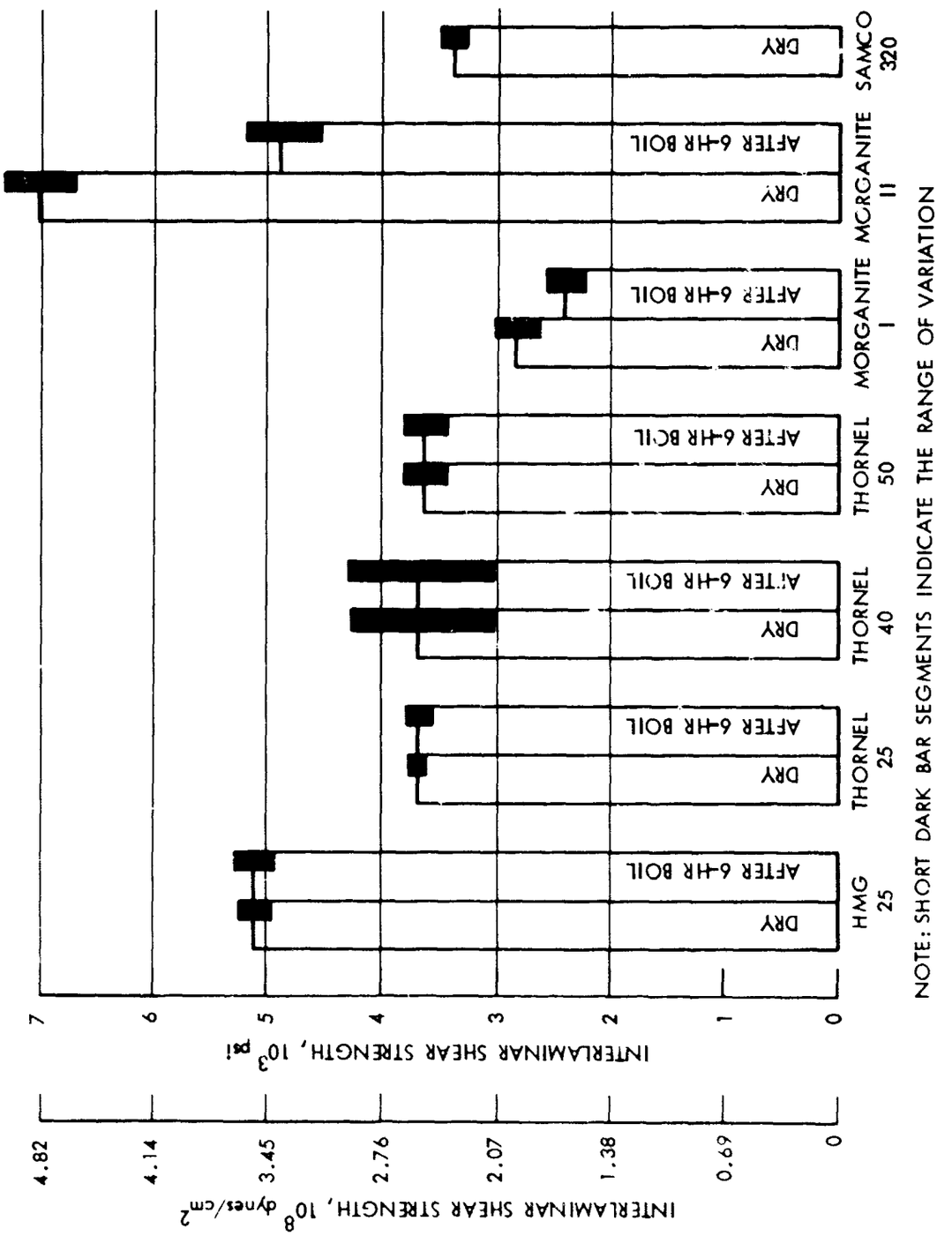


FIG. 6 TRANSVERSE STRENGTHS
Uniaxial Graphite Composites



NOTE: SHORT DARK BAR SEGMENTS INDICATE THE RANGE OF VARIATION

FIG. 7 INTERLAMINAR SHEAR STRENGTHS
Graphite Fiber Composites Short Beam Method;
Average Values from NOL Ring Specimens

FIG. 8 SINGLE WHISKER TREATED GRAPHITE FIBER

FIG. 9 SINGLE GRAPHITE FIBER PULLED FROM BUNDLE BEFORE AND AFTER WHISKER TREATMENT

FIG. 10 WHISKERED GRAPHITE FIBER (SILHOUETTE PHOTOMICROGRAPH)

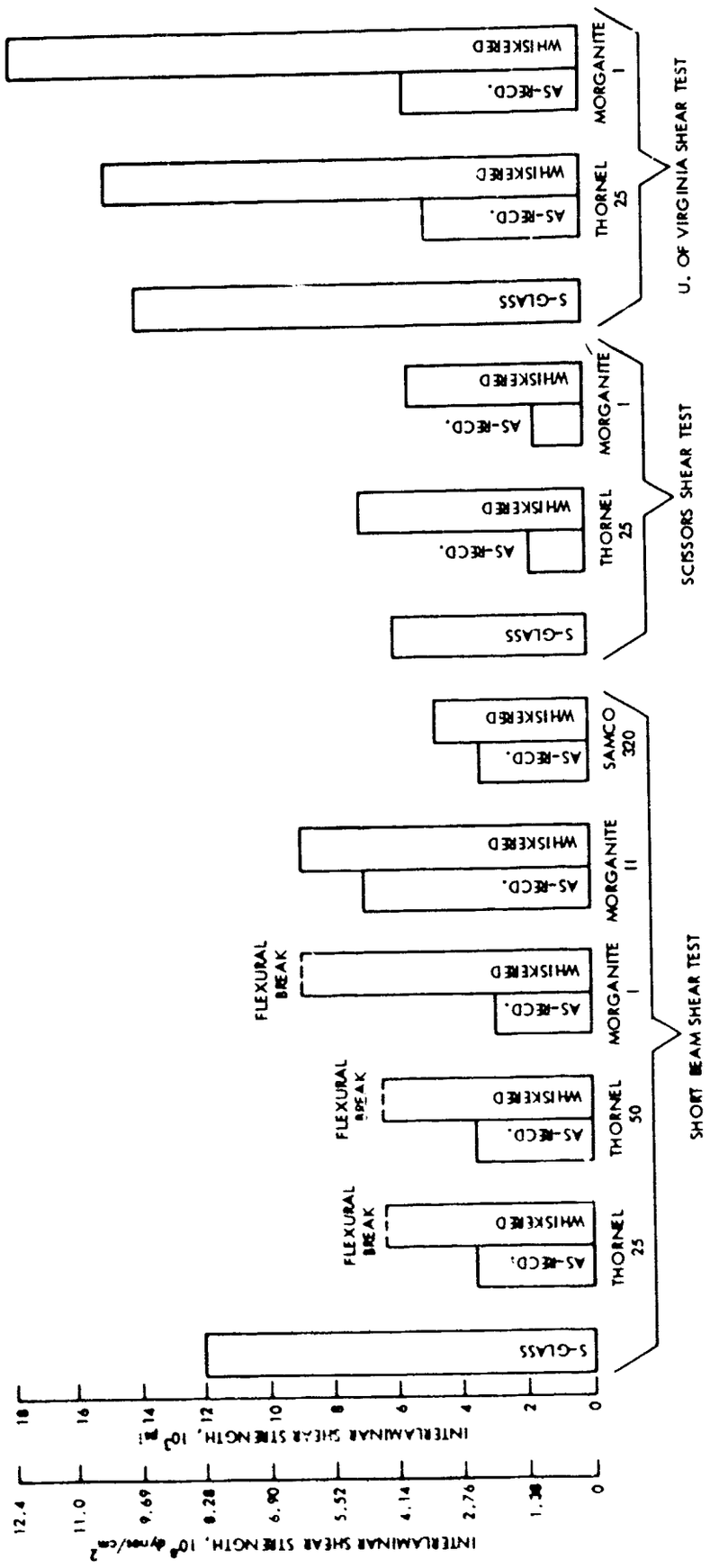


FIG. 11 INTERLAMINAR SHEAR STRENGTHS
Whiskered Graphite Fiber in Composites

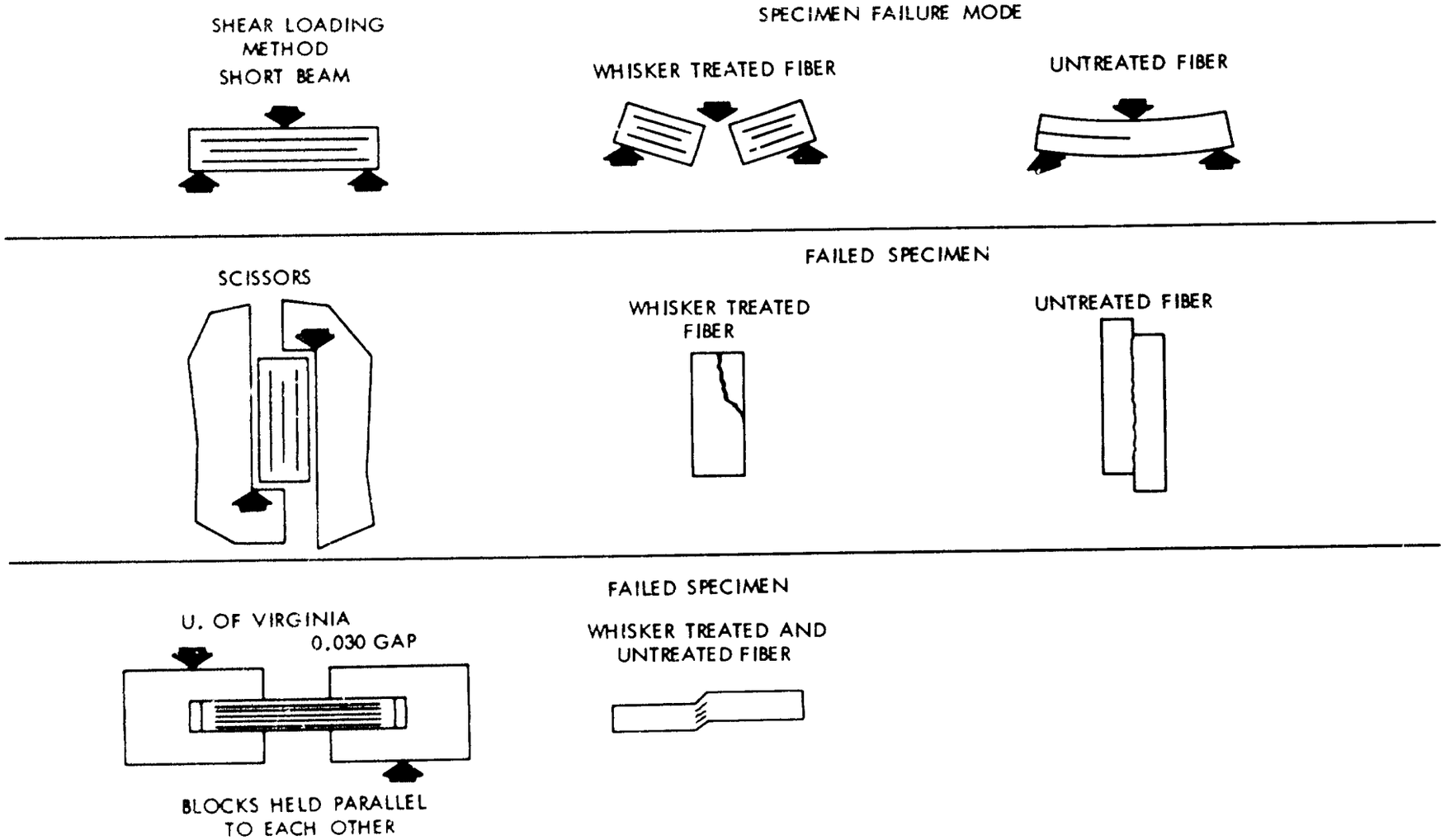


FIG. 12 SHEAR TEST SCHEMATICS

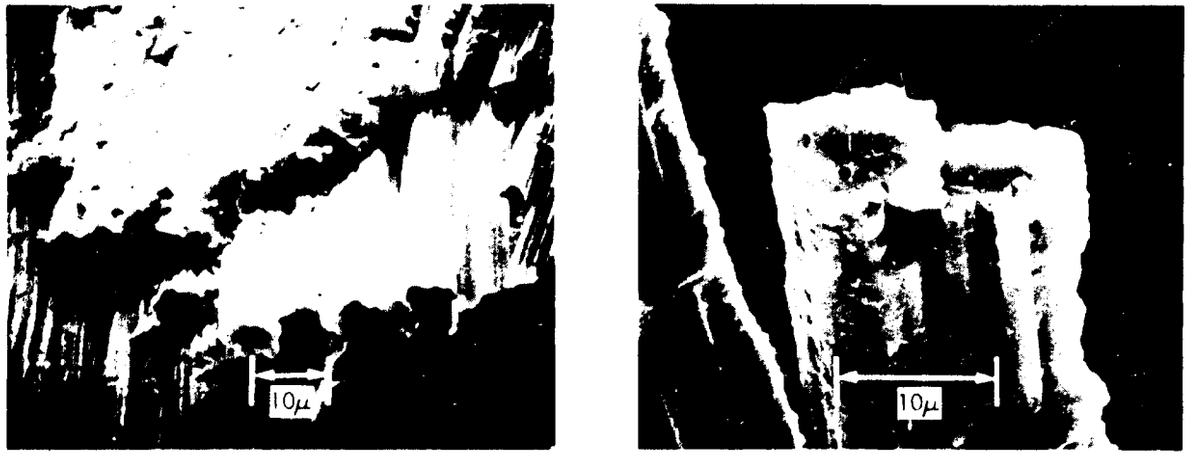


FIG. 13 UNWHISKERED FIBER COMPOSITE - VYB CARBON FIBER
Fracture Plane Indicates Good Bonding

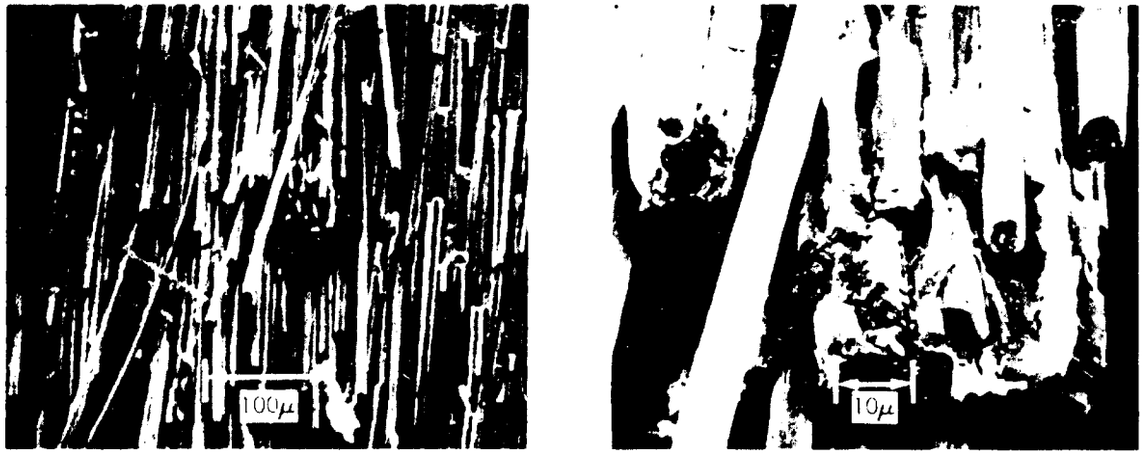


FIG. 14 UNWHISKERED FIBER COMPOSITE - RAE GRAPHITE FIBER
Fracture Plane Indicates Poor Bonding

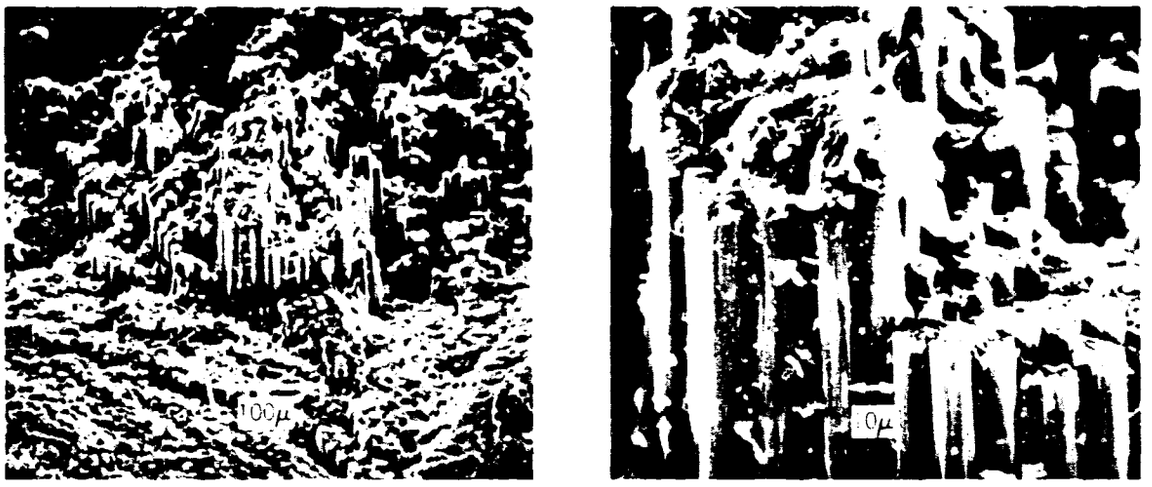
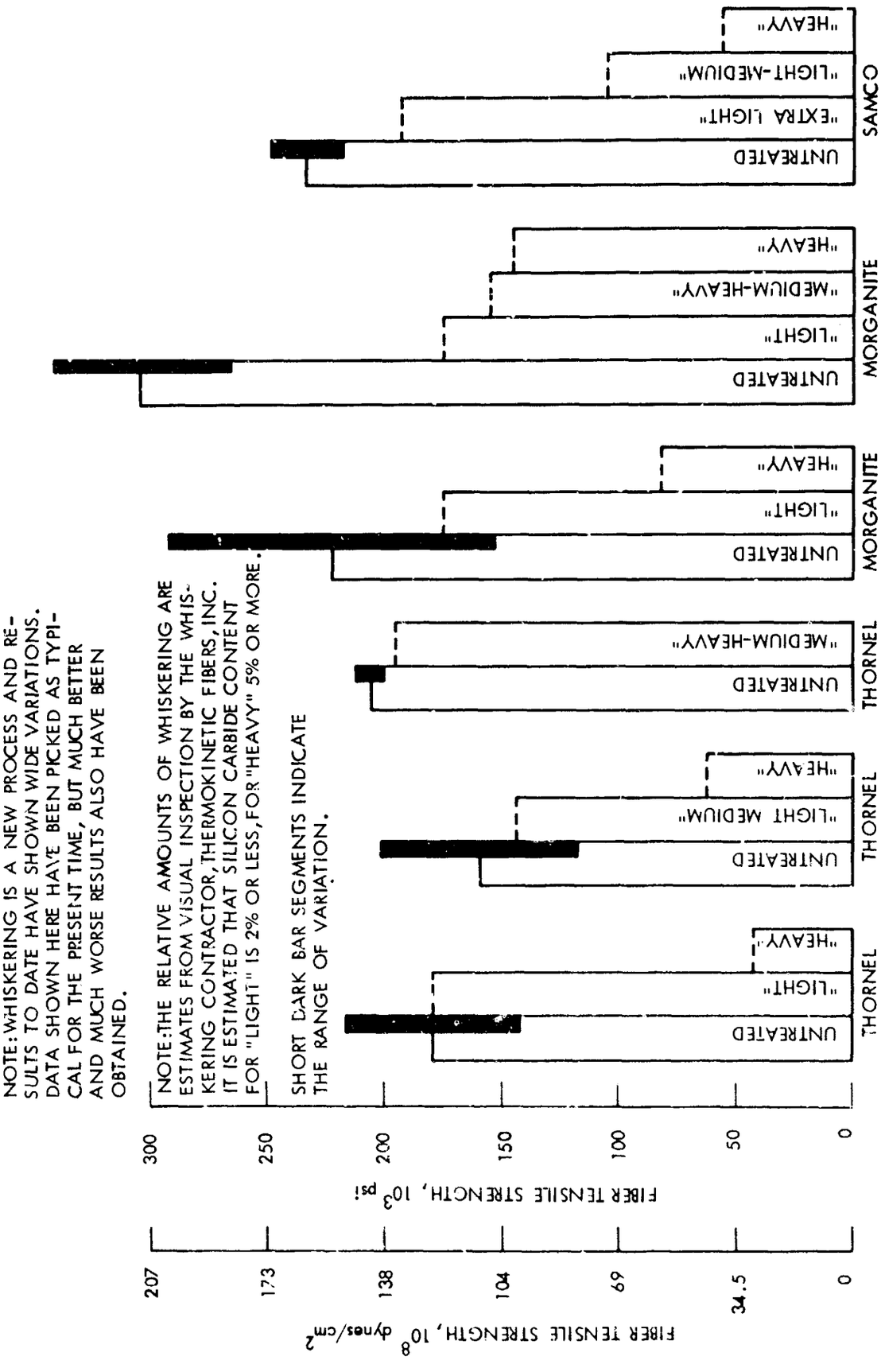


FIG. 15 WHISKERED FIBER COMPOSITE - RAE GRAPHITE FIBER
Broken Surface Indicates Good Bonding



NOTE: WHISKERING IS A NEW PROCESS AND RESULTS TO DATE HAVE SHOWN WIDE VARIATIONS. DATA SHOWN HERE HAVE BEEN PICKED AS TYPICAL FOR THE PRESENT TIME, BUT MUCH BETTER AND MUCH WORSE RESULTS ALSO HAVE BEEN OBTAINED.

FIG. 16 GRAPHITE FIBER TENSILE STRENGTHS AFTER WHISKERING
A Compilation of Strand and Filament Data

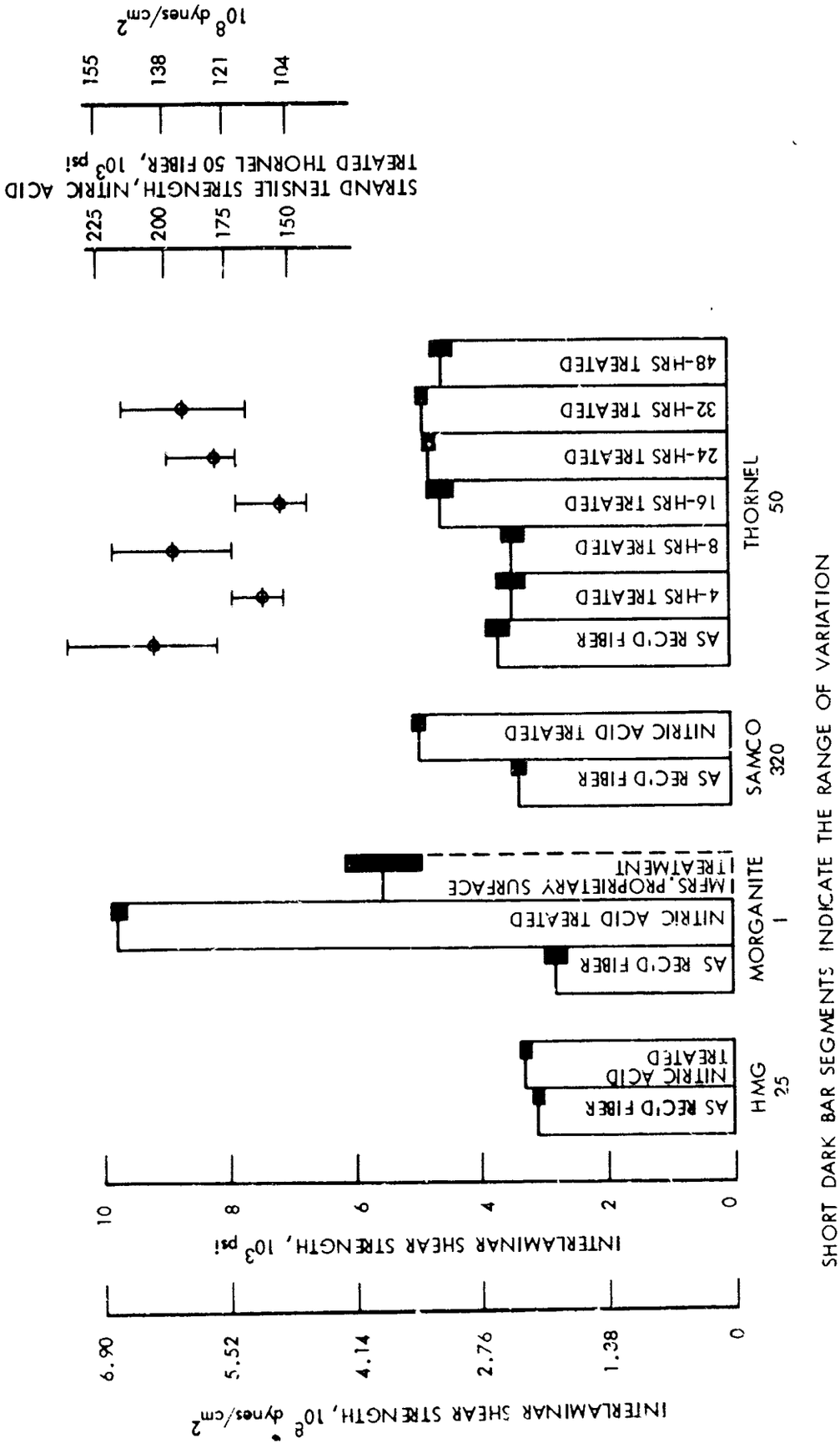
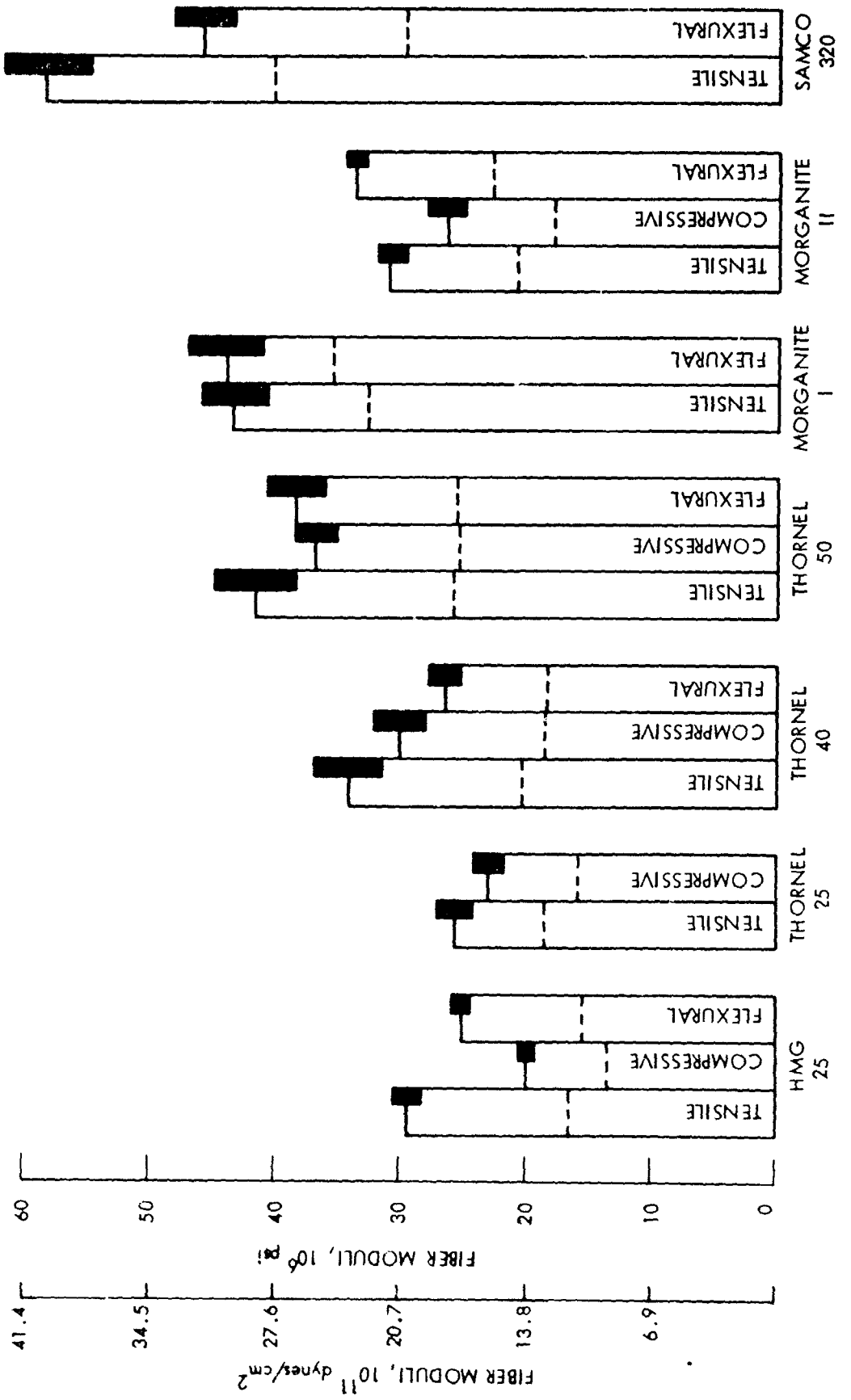


FIG. 17 INTERLAMINAR SHEAR STRENGTHS AND STRAND TENSILE STRENGTHS
Nitric Acid Treated Graphite Fibers in Composites



SHORT DARK BAR SEGMENTS INDICATE THE RANGE OF VARIATION

FIG. 18 GRAPHITE FIBER MODULI
Average Calculated Values from Tensile, Compressive, and Flexural Tests of Unidirectional Graphite Fiber Composites.

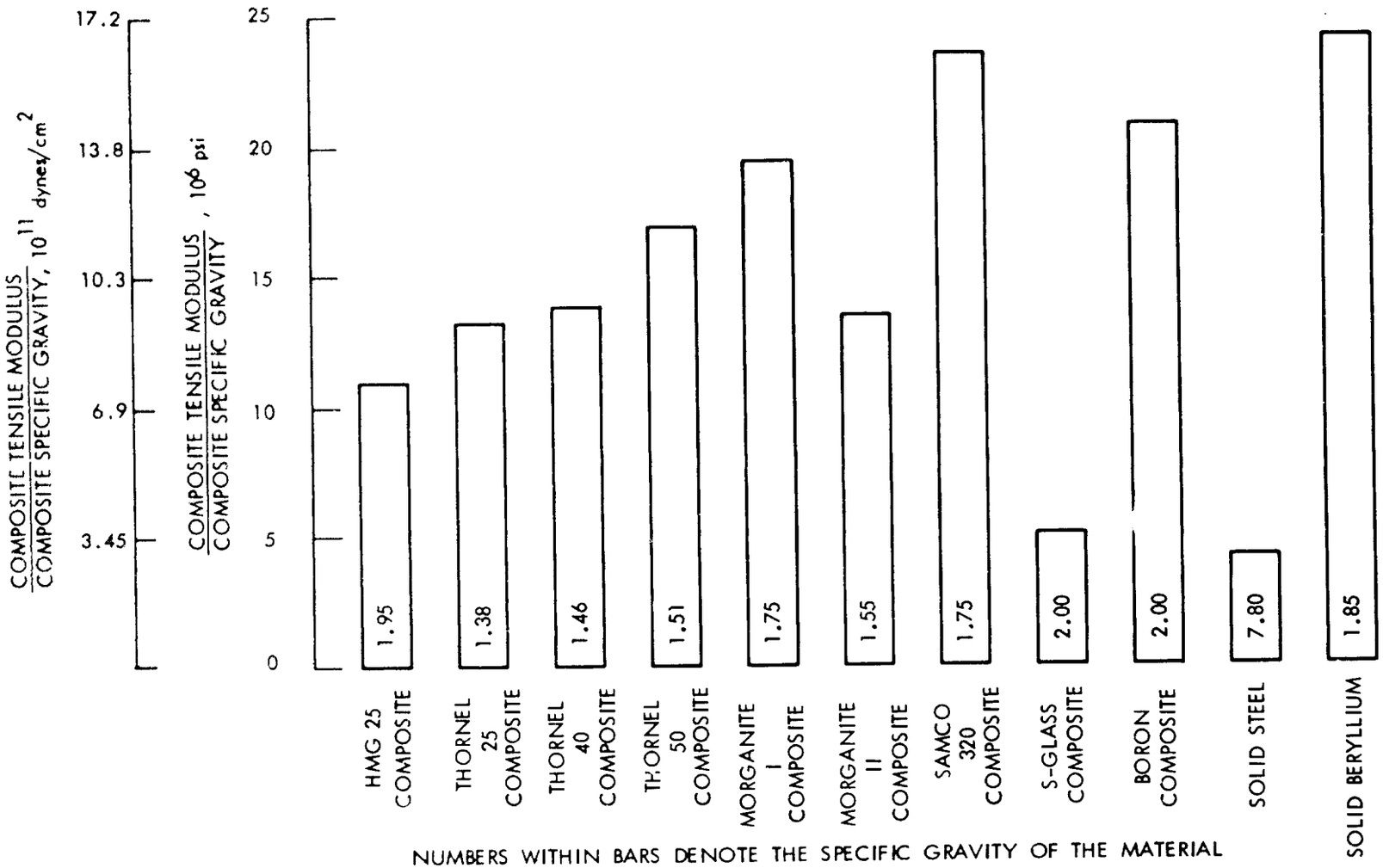


FIG. 19 AVERAGE SPECIFIC MODULI OF SELECTED MATERIALS
Composite Moduli Measured in Fiber Direction

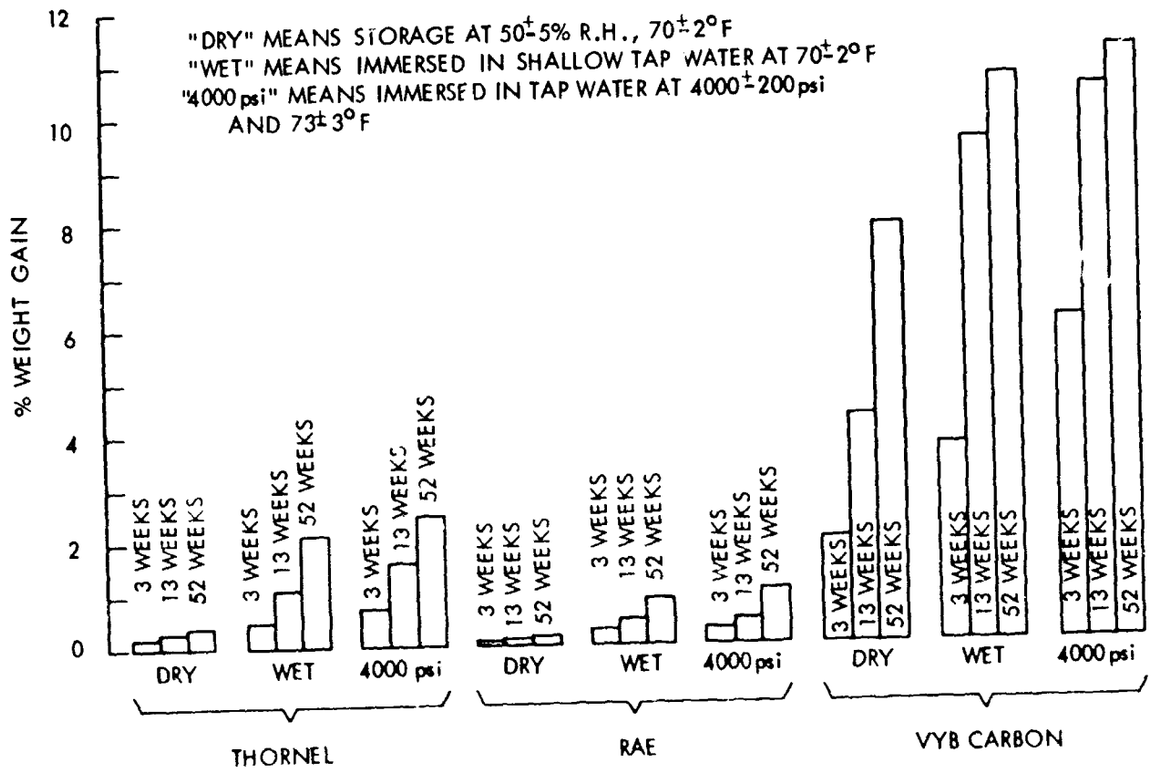
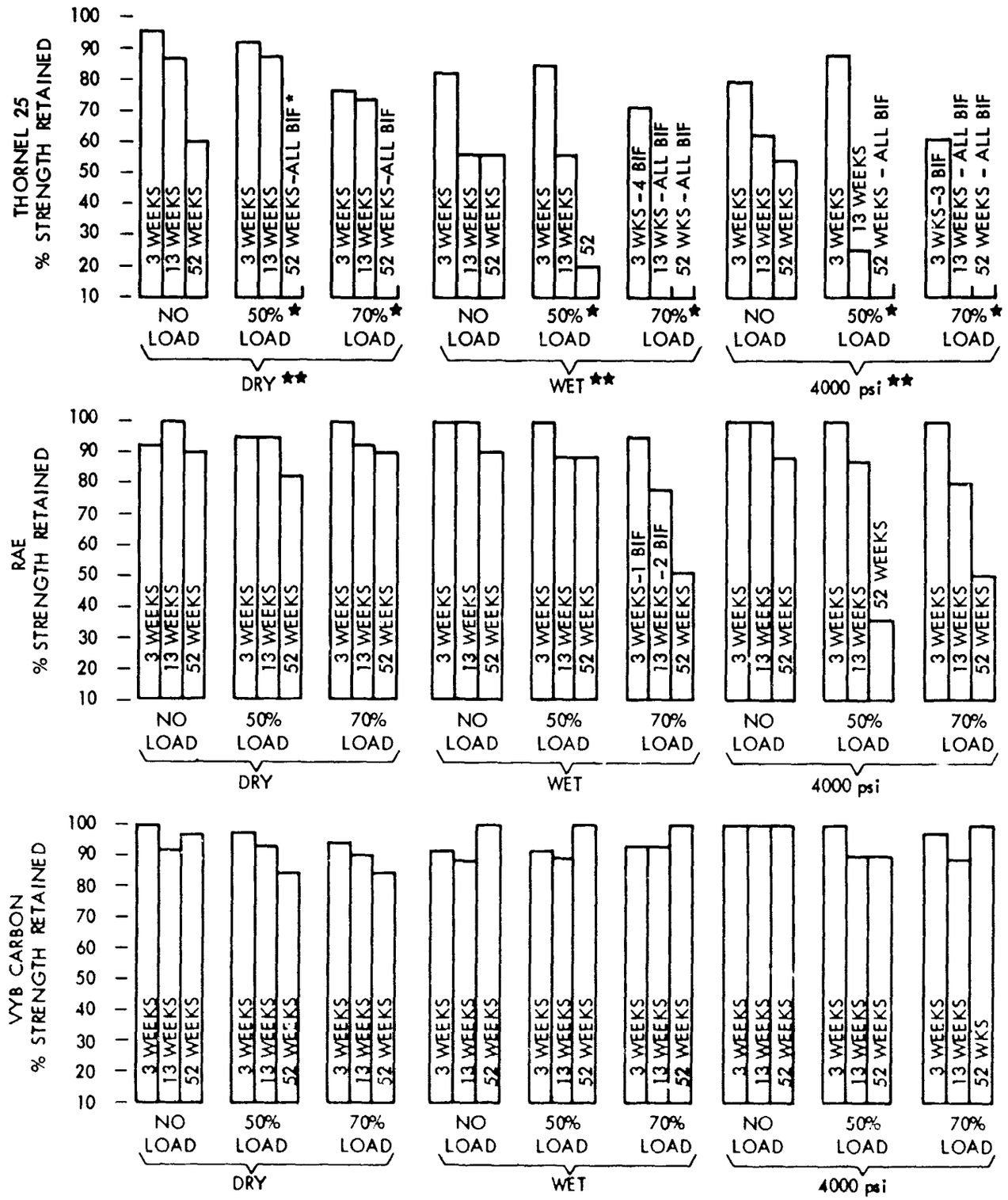


FIG. 20 LONG TERM WEIGHT GAINS AFTER VARIOUS EXPOSURES
Carbonaceous Fibers/2256:0820 Resin; Unidirectional Composites

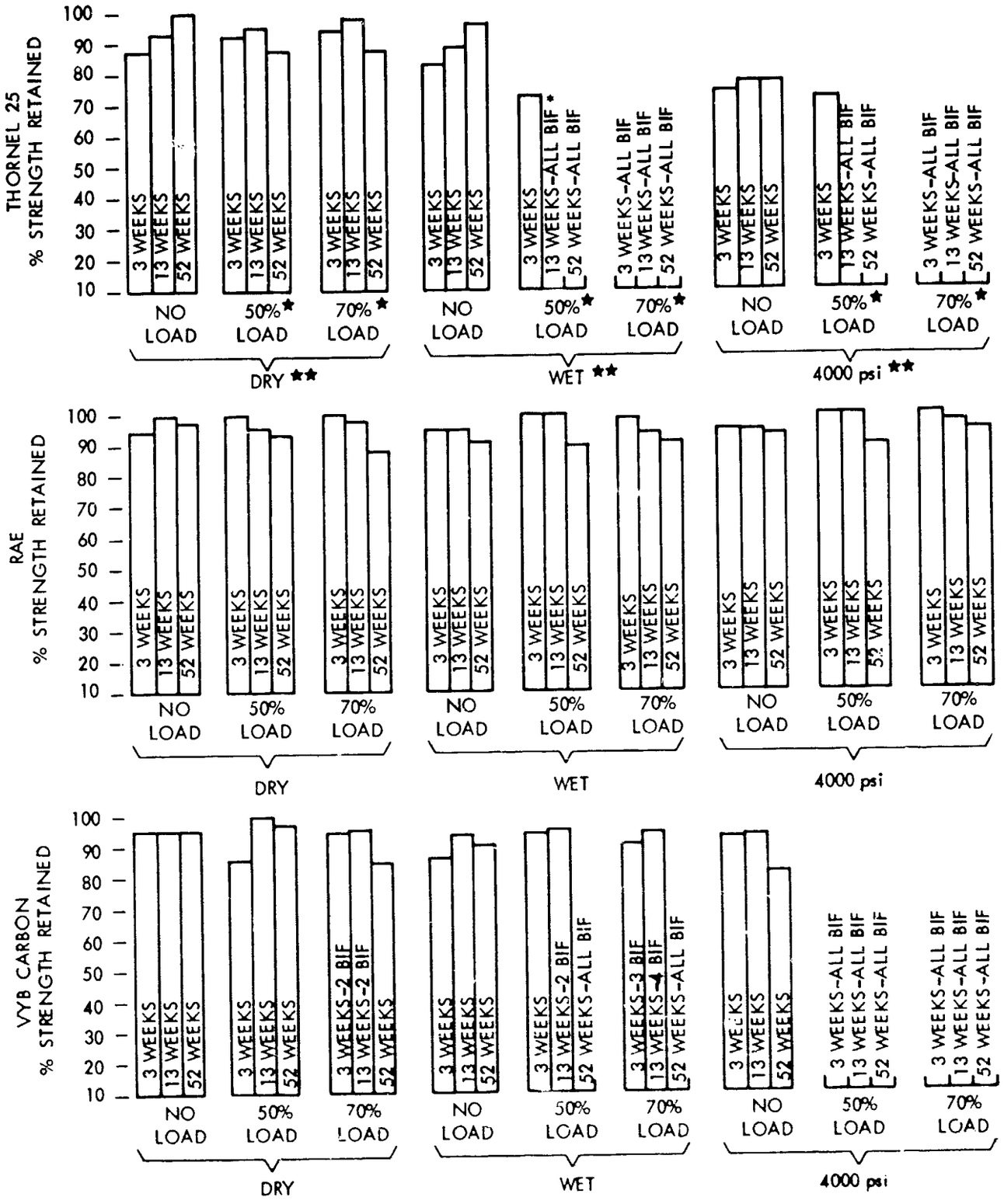


* BIF - BROKE IN FIXTURE

★ PERCENTAGES OF AVERAGE ULTIMATE DRY FAILURE LOADS FOR THE SPECIFIC COMPOSITES

★★ FOR DEFINITIONS OF THESE TERMS SEE FIG. 20

FIG. 21 LONG TERM FLEXURAL STRENGTH RETENTIONS OF COMPOSITES AFTER VARIOUS EXPOSURES
Carbonaceous Fibers / 2256:0820 Resin Unidirectional Composites

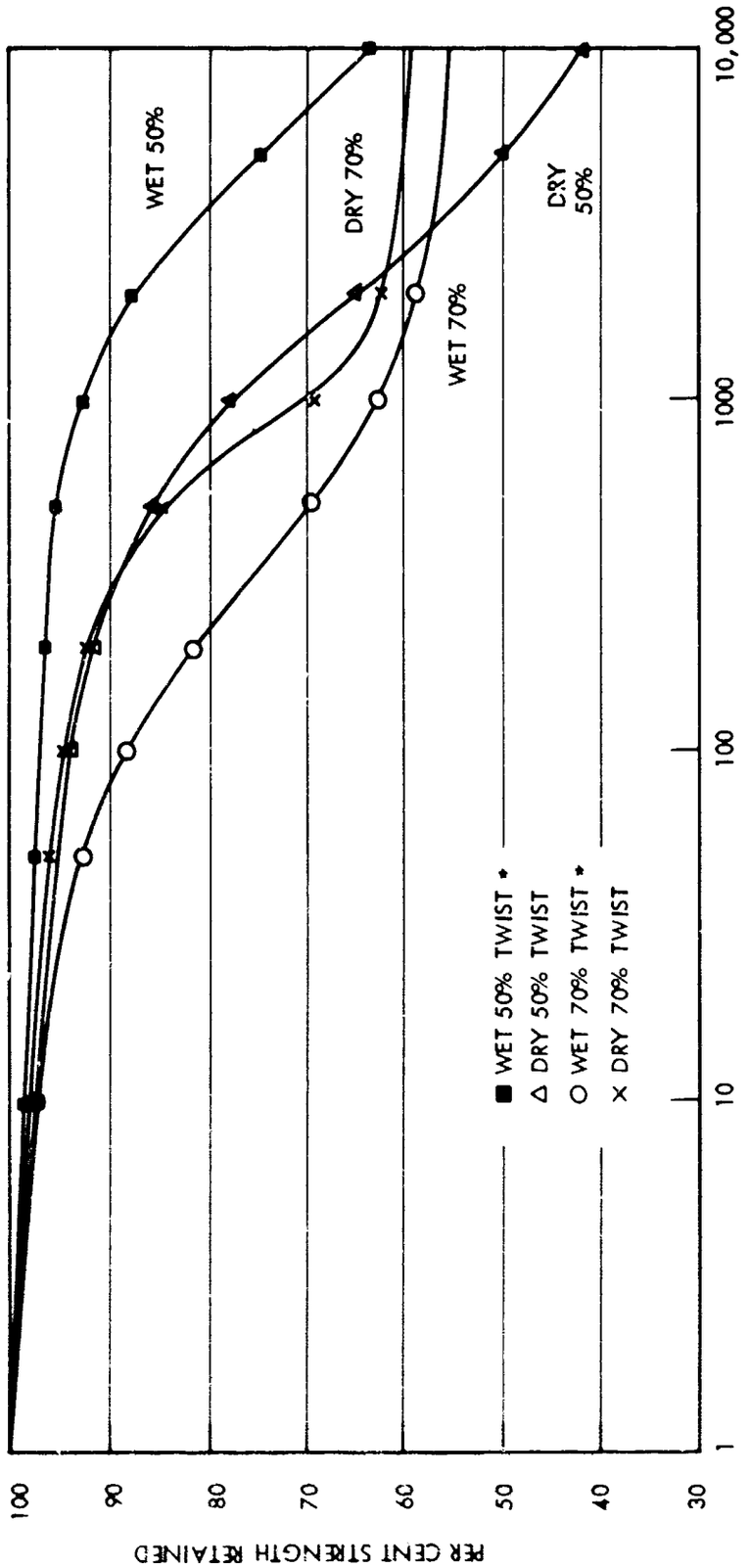


• BIF - BROKE IN FIXTURES

★ PERCENTAGES OF AVERAGE ULTIMATE DRY FAILURE LOADS FOR THE SPECIFIC COMPOSITES

★★ FOR DEFINITIONS OF THESE TERMS SEE FIG. 20

FIG. 22 LONG TERM SHEAR STRENGTH RETENTION OF COMPOSITES AFTER VARIOUS EXPOSURES
Carbonaceous Fibers / 2256:0820 Resin Unidirectional Composites



* TWISTS NOTED ARE PERCENTAGE OF ANGULAR TWIST NECESSARY TO BREAK CONTROL SPECIMENS

FIG. 23 MULTI AXIAL FATIGUE STRENGTH RETENTION
VYB Carbon Roving/2256:0820 Resin Composite

APPENDIX A

DESCRIPTION OF TEST METHODS

Method 1, Ultimate Tensile Strength--Individual Filament

A single filament was carefully extracted from the yarn (1440 twisted filaments) or the tow (10,000 parallel filaments) and laid across and in two molten wax pools atop a fixed and a movable post spaced 2.5 cm apart. The wax was allowed to cool and harden. The movable post was moved away from the fixed post at a speed of 0.100 cm/minute. The fixed post was attached to a load cell. An X-Y recorder recorded the output from the load cell and indicated the tensile load on the filament. Stress was calculated from the breaking load and the manufacturers' advertised nominal fiber cross-sectional area.

Method 2, Transverse Properties

Test	Specimen	Loading Speed
Tensile modulus ultimate strength	Straight bar 6.30 cm long x 1.26 cm wide	0.125 cm/minute
Flexural modulus ultimate strength	Straight bar 6.30 cm long x 1.26 cm wide	0.125 cm/minute
Compressive modulus ultimate strength	Straight bar 6.30 cm long x 1.91 cm wide	0.125 cm/minute

Thickness of all specimens was 0.242 to 0.254 cm.

Flexural specimen was given 4-point loading at 16:1 span/depth ratio.

Compressive specimen was held in a fixture to prevent buckling; 0.25 cm of the specimen extended beyond the fixture and was loaded through a ball and socket joint.

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13. ABSTRACT

Carbon and graphite fibers are now available with a range of physical properties; the graphite fibers are stiffest and strongest with moduli to 37 x 10^{11} dynes/cm^2 (54 million psi) and strengths to 28 x 10^9 dynes/cm^2 (400,000 psi). A total of 17 types of carbonaceous fibers was bonded with epoxy resins into unidirectional composites and tested for physical properties. Composite moduli and tensile strengths showed effective translation of fiber properties to composite properties, but shear strengths were low, ranging from 2.0 to 4.8 x 10^8 dynes/cm^2 (2900 to 7000 psi). These low shear strengths generally limited flexural and compressive strengths to relatively low values. A fiber treatment to grow silicon carbide whiskers and deposites on the fibers has given shear strengths up to 12.4 x 10^8 dynes/cm^2 (18,000 psi), but with fiber tensile strength reduction of 5 to 70%. Long-term water exposure of stressed composites showed varying results, depending on fiber and exposure condition.

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Security Classification

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Graphite Fiber Composites						
Reinforced Plastics						
Strengths and Moduli						
Silicon Carbide Whiskers						
High Modulus Structural Material						